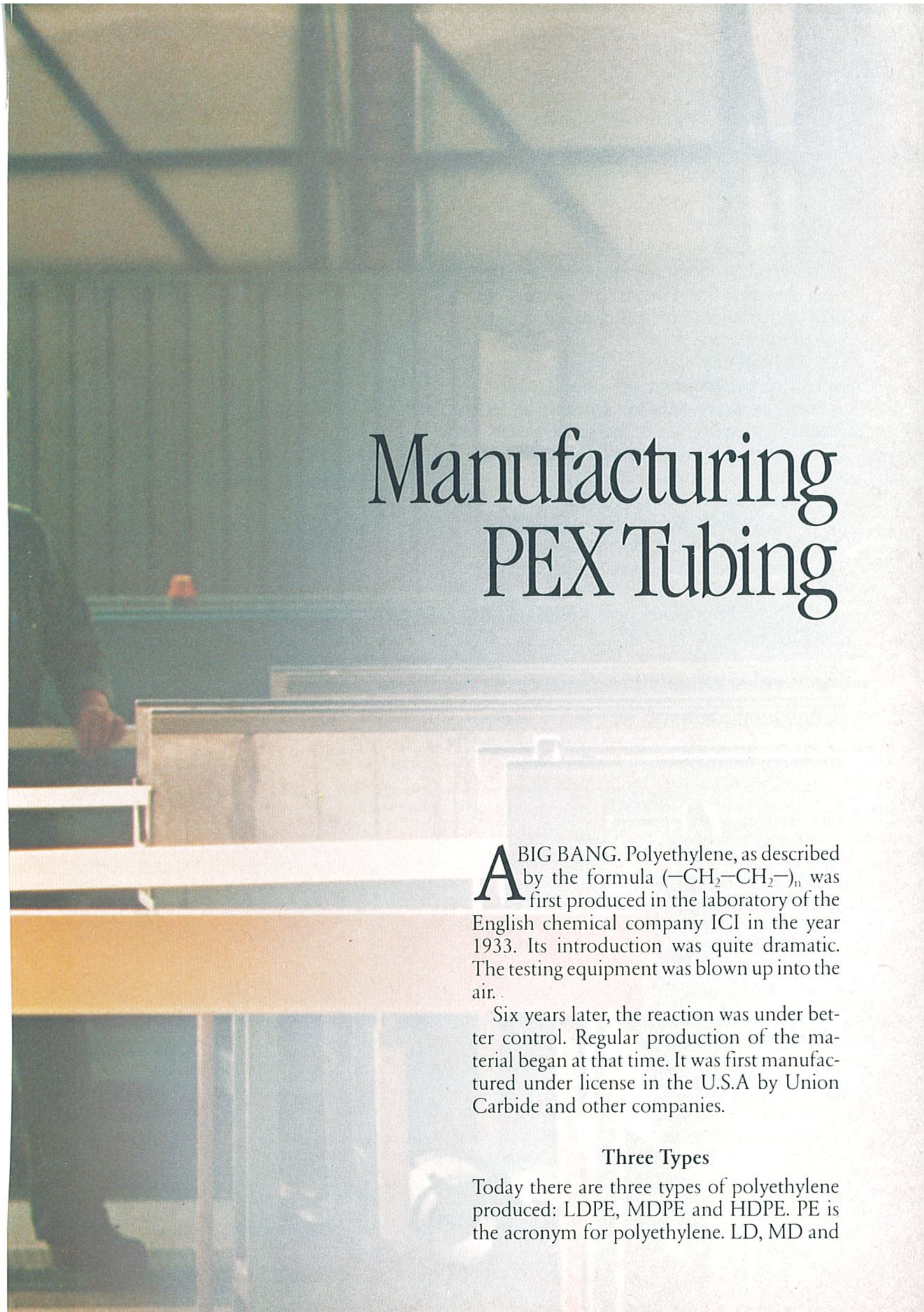


Manufacturing PEX Tubing

A BIG BANG. Polyethylene, as described by the formula $(-CH_2-CH_2-)_n$ was first produced in the laboratory of the English chemical company ICI in the year 1933. Its introduction was quite dramatic. The testing equipment was blown up into the air.

Six years later, the reaction was under better control. Regular production of the material began at that time. It was first manufactured under license in the U.S.A by Union Carbide and other companies.

Three Types

Today there are three types of polyethylene produced: LDPE, MDPE and HDPE. PE is the acronym for polyethylene. LD, MD and

HD stand for low density, medium density and high density. The division of the three types is made according to these ranges:

LDPE: 56.8–57.8 lb/ft^3 (0.910–0.925 Mg/m^3)
MDPE: 57.9–58.6 lb/ft^3 (0.926–0.940 Mg/m^3)
HDPE: 58.7–60.0 lb/ft^3 (0.941–0.965 Mg/m^3)

Wirsbo-PEX tubing is produced from an HD polymer with an extra-high molecular weight and a density of about 59.3 lb/ft^3 (0.95 Mg/m^3). This factor is decisive in determining the properties of the final product because the crystallinity of the polymer is directly related to its density.

"Crystallinity" refers to the regular arrangement of molecules in the overall structure caused by strong adhesive forces in the material. The opposite of that, the irregular arrangement of the molecules, is referred to as a material's amorphous state.

Both types of structures can be seen in the schematic illustration shown in Plate 8:2.

The greater the density, the higher the degree of crystallinity and, as a result, the greater the strength of the plastic. The extent of crystallinity in the polymer that Wirsbo uses for its PEX tubing is 90 percent.

The X Stands for Cross-linked

The X in the acronym PEX refers to cross-linking. This term is used to describe the chemical linking of the PE molecules into a three-dimensional network.

The technique that Wirsbo uses to achieve the cross-linking in its tubing material is based upon a process developed by the German inventor, Thomas Engel. The cross-linking that is attained using that method is the basis for the outstanding characteristics of Wirsbo-PEX tubing.

Monitoring the Raw Material

Before the manufacturing process begins, the macromolecular raw materials and the additives, such as stabilizers, cross-linking agents (peroxide) and so on, are subjected to thorough monitoring procedures. Ratings such as the melting index, homogeneity and density are determined in Wirsbo's own laboratory.

A portion of the raw material that is found to be satisfactory is then put into the laboratory mixer. From there it is taken to the testing

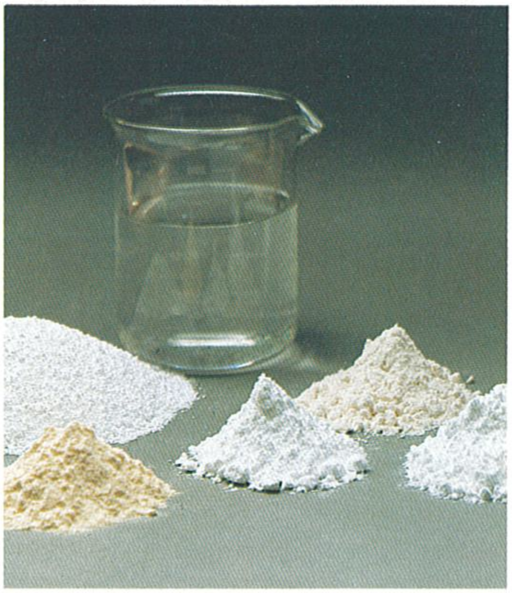
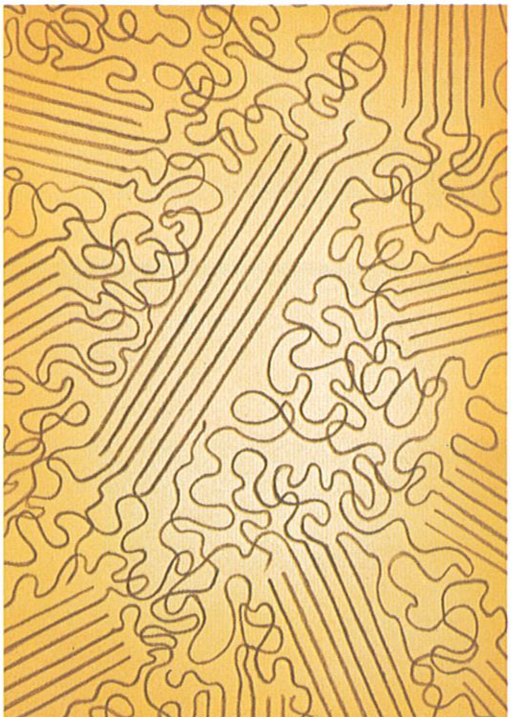


Plate 8:1

***The raw materials** used to make Wirsbo-PEX tubing.*

***The structural arrangement** of the polyethylene (HDPE) chain molecule. The crystalline (regular) and amorphus (irregular) areas are very clearly defined.*

Plate 8:2



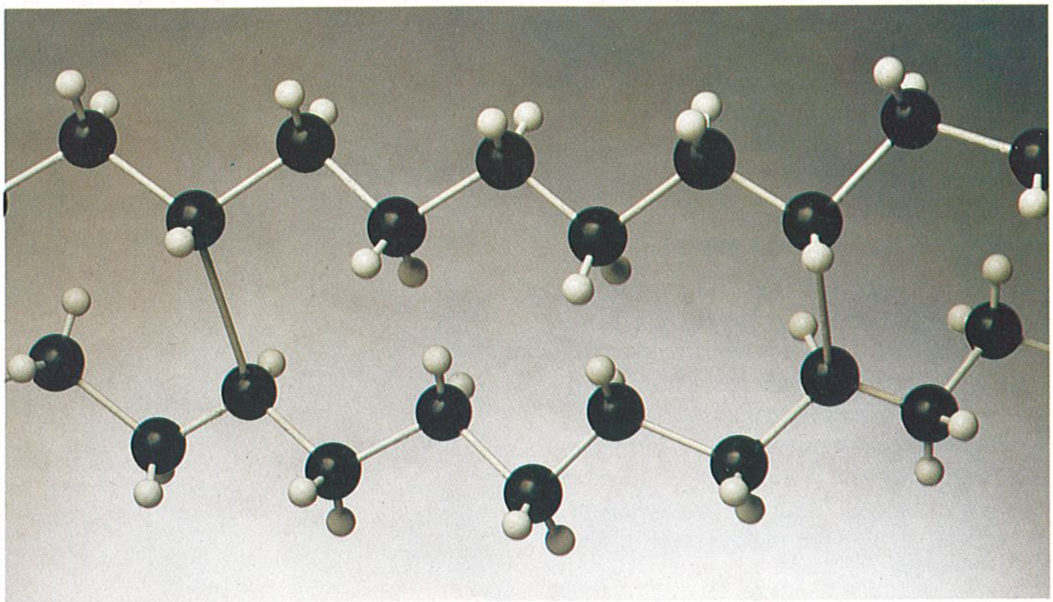


Plate 8:3

The molecular structure *of cross-linked polyethylene. The chain molecules are linked to each other by molecule bridges.*

Plate 8:4

Wirsbo determines *the exact amounts to add for every shipment of raw material.*

track or "pilot plant" as it is sometimes called. Regular production begins only after it has been determined from an actual finished piece of tubing that the composition of the material, as well as the technique used to produce it, is correct.

440 Pounds for 5,000 Feet

The raw polyethylene that Wirsbo procures in powder form is strained to get just the size of grain desired. Then it is weighed electronically and combined with the additives in a mixer using an elaborate technique.

The mixture is transferred to a 440-pound (200 kg) container. After being marked according to the type of tubing that will be produced from it, it is set aside in a holding room for a few days.

Finally the mass is fed from the containers into the extruders. 440 pounds (200 kg) of the powder is enough to produce about 5000 feet (1500 m) of 3/4-inch (20 mm) Wirsbo-PEX tubing with 3/32 inch (2 mm) thick walls.

Cross-linking in its Amorphous State

The tubing is given its shape by an extruder that, in general terms, takes up the raw material in powder form at one end and, by applying heat and a great degree of pressure, forces the tubing out through a ring-shaped opening at the other.

Cross-linking also takes place while the tubing is being formed. At this time, it might be well to mention an important difference between Wirsbo-PEX tubing and other tubing made of cross-linked polymers. The cross-linking takes place while the material is in an amorphous state, that is, at a temperature above that at which the crystals melt. As a result, there are no crystalline areas present that could have a negative effect upon the cross-linking process. The linking can take place evenly and without hindrance.

Searching for Defects Electrically

As the amorphous material emerges from the molding machine, it is almost transparent. While still in that state, it is subjected to monitoring equipment that employs an electrical field to continuously analyze the finished

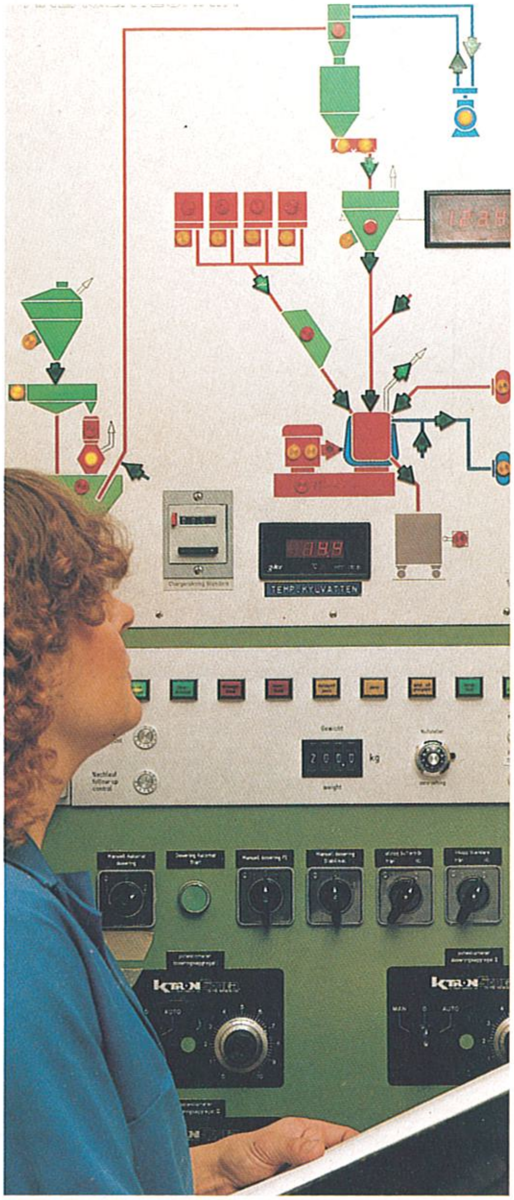


Plate 8:5

Plate 8:5. *The control unit of the automatic raw-material dispensing unit used for Wirsbo-PEX tubing.*

Plate 8:6. *The raw-material containers are exchanged during the ongoing production process.*

Plate 8:7. *Analysis of the temperature-dependent behavior of plastics (melting point and so on). Method: Differential Thermal Analysis (DTA).*

Plate 8:6

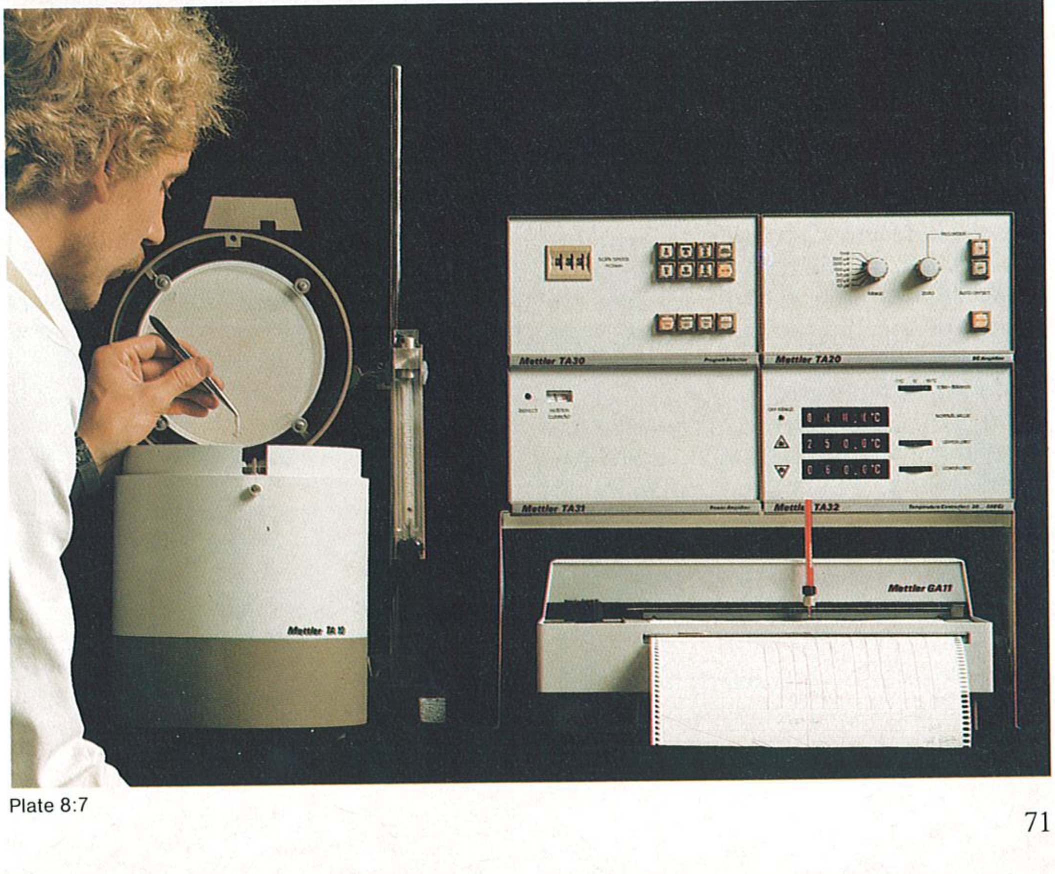


Plate 8:7

tubing. It uses a field intensity of 12,000 Volts for every 1/16th inch (7,500 volts for every millimeter) of wall thickness. Every disturbance in the electrical field that might be caused by a deviation in wall thickness or by the incorporation of air or dirt immediately sets off an alarm. Besides monitoring for thickness and homogeneity, any gaseous remains in the material are removed at this time.

Calibrating equipment and a cold water bath follow right after the monitoring equipment. It is at this spot that the tubing material crystallizes and takes on its characteristic, milky white color. Right after that, the tubing is printed with detailed information, cut for length and wrapped into a coil ready for shipping.

Entries are made in a daily plant journal for every coil showing the date, time of day, the responsible operator, and other similar types of information. Other items are also entered into this journal, such as the type of material being used at any given time and any changes in extruder dies.

Not One Coil is Overlooked by Wirsbo

The Wirsbo laboratory collects a sample from each coil and then puts them through an almost painfully exacting battery of production tests and checks. Wirsbo has set up standard tolerances for inside and outside diameters and for wall thicknesses that are lower than the tolerances normally allowed for plastic tubing. If there is a variation of .0008 inches (0.02 mm) outside of the set tolerances for any of the three measurements mentioned, the whole coil is considered to have failed the test. It is sawed to pieces.

This "finickiness" has a solid practical purpose. If the person installing Wirsbo piping is to work as fast as Wirsbo always insists he can, the pipe fittings must sit exactly right the first time.

Cross-linking: Not Too Much, Not Too Little

The properties of tubing made of cross-linked polyethylene are determined mainly by the degree of cross-linking they undergo, that is, by the proportion of molecule chains that are

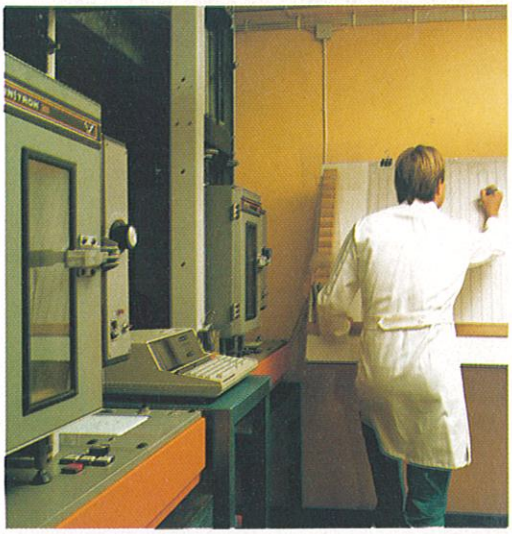


Plate 8:8

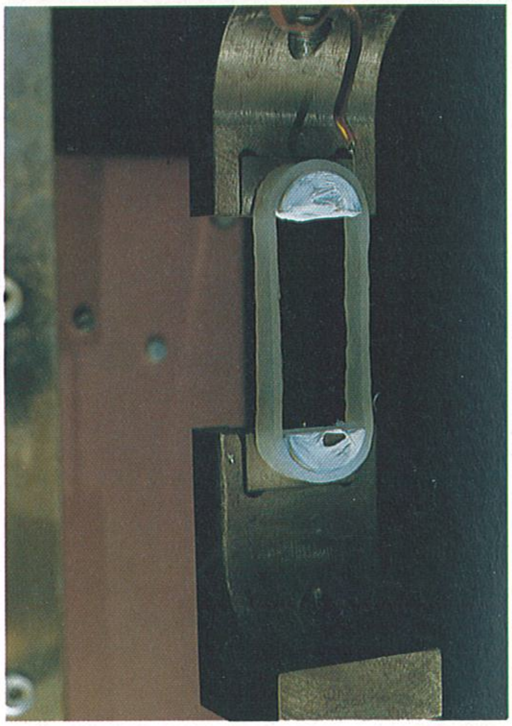


Plate 8:9

Plate 8:8. *Production monitoring in the Wirsbo factory. Every value is recorded in a journal and archived.*

Plate 8:9. *Determining the degree of cross-linking in tubing materials using the quick method: testing for tensile strength after heating to a temperature of 310° F. (155° C.). (Plant photo: Studsvik Energiteknik AB).*

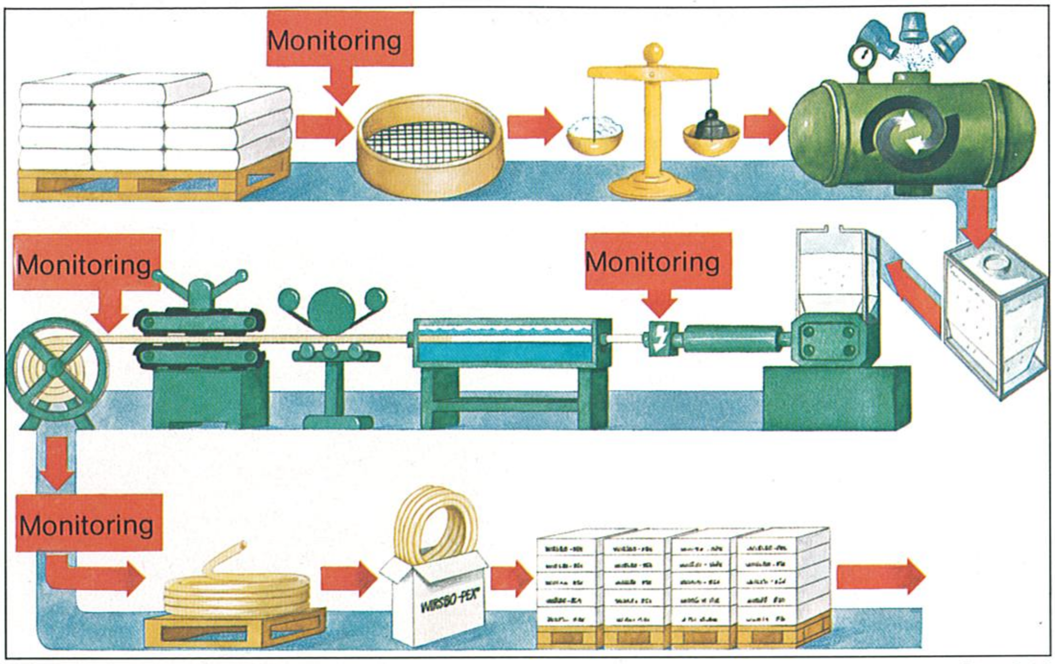


Plate 8:10

Plate 8:10. *A simplified outline showing the production of Wirsbo PEX tubing. Monitoring is done at various places along the production line.*

Plate 8:11. *Monitoring the dimension tolerances of finished PEX tubing.*

Plate 8:11

linked to each other. This proportion has to be well balanced. If it is too small, the material will be more like noncross-linked PE with its limited durability at high temperatures. If it is too large, the material will quickly become brittle.

The degree of cross-linking in Wirsbo-PEX tubing should lie between 70 and 90 percent. The Wirsbo laboratory checks this out using two different methods. The first employs a fast but very reliable mechanical method (our own patent) and the second uses chemical analysis based upon ASTM and DIN standards. The second method is also used for calibrating the mechanical test.

Pressure Test

Once a week, Wirsbo takes five samples from each production line for a pressure test. The samples are placed in 203°F. (95°C.) water, filled with water and kept under excess pressure for 170 hours.

For example, a pipe with a nominal pressure rating of 87 psi (6 bar) is tested by Wirsbo at an internal pressure of 145 to 174 psi (10 to 12 bar). The result is a hoop stress on the tubing wall of 667 psi (4.6 N/mm^2). This is a value very close to the actual strength of the material. There is a good reason why Wirsbo works with such high values in these tests. Wirsbo wants to apply the same stress in the short-term tests as used in the compilation of the time-to-failure graphs. This approach provides an additional guarantee of quality. Wirsbo also conducts similar tests which last for 1000 hours although less frequently.

In accordance with valid standards, one-hour pressure tests and even burst-pressure tests are also performed. It is Wirsbo's view that these short-term tests are of no significance and should be omitted if certain relevant raw-material tests are performed.

Dimensional Change Test

The samples are taken from each production line three times a week. They are measured in the most precise way possible. Then they are heated in a 248°F. (120°C.) oven for one hour. Finally they are measured again after they have been taken out and cooled off.

This test gives Wirsbo an insight into the

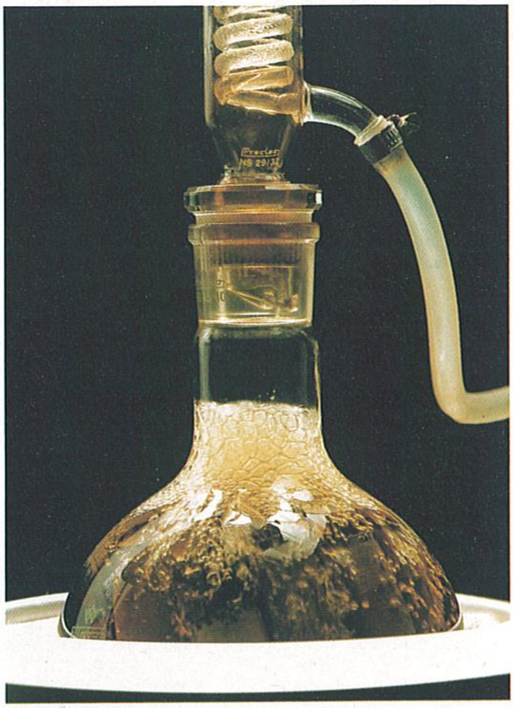


Plate 8:12

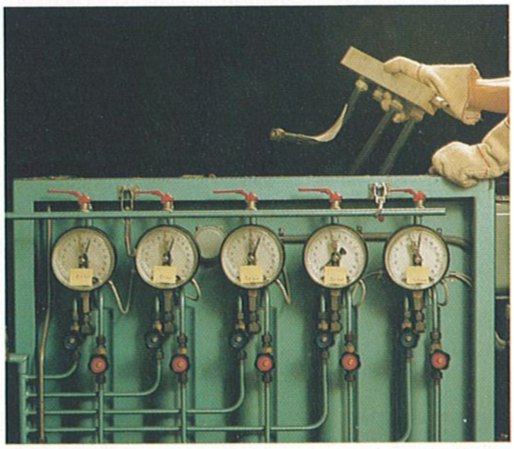


Plate 8:13

Plate 8:12. *Chemical determination of the degree of cross-linking using the standard methods.*

Plate 8:13. *Pressure test on bent tubing. This test is intended to provide proof that no ruptures would occur within the durability range specified for the tubing.*

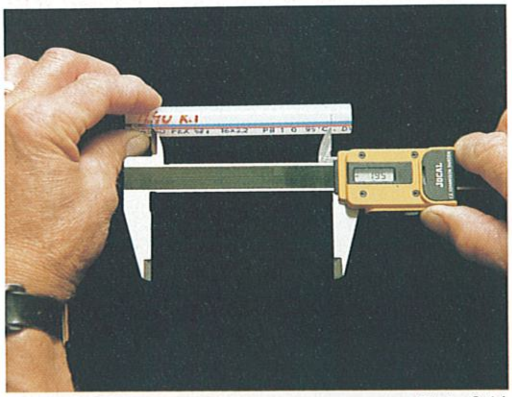


Plate 8:14

Plate 8:15

Plate 8:14. Elongation test. The change in length of the sample is measured after it has been heated in an oven.

Plate 8:15. Official quality monitoring. Dr. Stagge of the MPA Darmstadt entering the results gained from a random sampling into a log book.

amount of internal stress in the material used for its tubing. Some testing standards allow 3 percent and others 5 percent as the upper limit of acceptable change in a tubing's length. Wirsbo has set its upper limit at 3 percent.

In the case of some materials, a higher percentage of change in its shape indicates a greater tendency towards breaking and rupturing.

The Watchful Eye

The production monitoring that is done in the Wirsbo plants should:

1. prevent the sale of defective tubing and
2. ensure that all tubing sold will meet the requirements of the official certification especially since it was based upon the results of tests conducted by Wirsbo.

In order for the internal production line monitoring to become as trustworthy as possible, it has been placed under the supervision of neutral testing boards. Two to three times a year, representatives from such boards in the United States, West Germany, Holland, Norway, France and Sweden visit the Wirsbo plants. What is even more, they appear unannounced. They inspect the specifications, journals and testing equipment and they take samples with them back to their laboratories for testing.

Quality has been an integral part of the Wirsbo tradition ever since the company was founded in the seventeenth century. It was in this spirit that such an extensive system of quality assurance was developed. The demands that Wirsbo places upon itself are high. Wirsbo strives to be the best. Anything less than that is not enough.

MORE ON CROSS-LINKING. Over the past years, the plastics industry has developed various cross-linking methods for PE molding or extrusion either alone or in combination with other plastic materials. Just because a plastic is given the generic name "PEX" does not mean that it contains the same material as all the rest that are known by the same name. Every manufacturer produces a version of PEX tubing that distinguishes itself from that produced by other

***Various brands** of PEX tubing.* Plate 8:16

manufacturers. These differences can be caused by the characteristics of the raw material, by the method used to produce the final product and by the degree of quality demanded.

In addition to the Engel process used by Wirsbo, there are other cross-linking methods in use.

Radiation Cross-linking

When normal PE tubing is bombarded by electrons, the energy produced by the rays sets the cross-linking action into motion. This process occurs at room temperature. The linking that takes place is concentrated on the areas in the material that are still amorphous at the time of the bombardment. Doing so does not appreciably reduce the degree of crystallinity achieved.

This relatively new method is best suited for small-sized tubing. It has, however, some drawbacks. One danger is that of holes forming in the tubing wall as the result of a possible electric breakthough during the bombardment. There is also a tendency toward uneven cross-linking or toward excessive cross-linking with brittleness as a possible result.

Silane Cross-linking

This process, which was developed by the Dow Chemical Company, is based upon saturating the PE macromolecule with silicon. Subjecting it to water vapor in the presence of a catalyzer activates the cross-linking process. The linking takes place through so-called siloxane bridges (—Si—O—Si—). These bridges are, however, weaker than normal C—C bonds.

Sioplas Method

This process takes place in two steps.

In the first step, the polymer is mixed with an organic silicon derivative. Under the influence of a peroxide and an antioxidant, it saturates the PE chain.

In the second step, the pretreated polymer and a catalyst is mixed with a PE base material. After this is done, the tubing is produced. The cross-linking takes place relatively slowly through the addition of water vapor and heat. It is determined by how well the water penetrates the tubing wall.

Monosil Method

This cross-linking technique, which was developed by the Maillefer and BICC companies, is similar to the Sioplas Method insofar as its chemical reaction is concerned. The difference is that only one step is required. It is used primarily for the manufacturing of cables and for tubing to be used in the middle temperature ranges.

Pont-à-Mousson Method

Under the PAM method, the tubing is shaped at the temperature at which peroxide reacts. This means that the cross-linking takes place in a fused-salt bath at a temperatue of 480° to 540°F. (250° to 280°C.).

When using this method, it does become a problem to preserve the shape and the surface characteristics of the tubing during the cross-linking stage because of the high temperatures used. Compared with the Engel process, it also requires a larger amount of peroxide.

AZO Method

The Swedish company, Uponor AB, uses AZO compounds (molecules with the grouping —N=N—) in its method for cross-linking PE. The tubing is formed at a temperature lower than that at which AZO unions occur. Then the temperature of the salt bath is raised up to that required for the reaction to take place. It is substantially higher than the temperature required for a peroxide reaction.

The PE that is being used in this case is one with a high density and a high molecular weight. The cross-linking takes place in a fused-salt bath in a way similar to the PAM method.

Other Peroxide Methods

Several recently developed methods all use peroxide for cross-linking. Peroxide is either mixed with the raw materials before extrusion or added by diffusion techniques during or after the extrusion process. In either case, the shape of the tubing must be carefully controlled during the cross-linking period when the temperature is increased. Because these methods are quite new, they are still undergoing further development.

UHF Cross-linking

Professor Menges of the Technological University at Aachen recently developed a new method for cross-linking polyethylene.

This method is based upon the fact that polarized substances absorb energy from a UHF (Ultra High Frequency) field. In this way, the peroxide disintegrates into radicals that can bring about cross-linking and it does so at a temperature that is lower than that normally required. The PE chain itself is not polarized and so does not absorb any energy.

It is also possible to add other polarized substances (carbon black) and to use otherwise "normal" peroxide that will form radicals through thermal decomposition. The mass, along with all its additives, is then extruded at as low a temperature as possible. After that, it is cross-linked in a UHF stage.

One advantage of this method is the even distribution of the energy throughout the material which results in an homogenous cross-linking effect.

The problems with this method are the search for suitable, nonpolarized additives for the tubing and the effect that remaining radicals might have.

This method is not yet ready for use in mass production.

A Comparison of Properties and Behavior

Material 1: *Cross-linked polyethylene (PEX) produced by the Engel method as further developed by Wirsbo.*
Material 2: *Polyethylene cross-linked by radiation.*
Material 3: *Polyethylene cross-linked by chemical methods other than the Engel method.*
Material 4: *Other polyolefin materials, such as PB, PP, PPC (PE).*

Property	Materials			
	1	2	3	4
Thermal stability at 203°F. (95°C.)	1	2	2–3	2–4
Time-to-failure up to 203°F. (95°C.)	1	2	2–3	2–4
Stress-crack resistance	1	2–4	1–2	2–4
Flexibility	1	2	2–3	2–4
Impact strength	1	1	1	3–5
Thermal conductivity	2	2	2	3–4
Elongation	4	4	4	3–4
Measurements, tolerances	1	2–4	2–4	2–4
Surface characteristics	1	2	2–5	2
Mechanical properties at room temperature:				
Short-term strength	2–3	2–3	2–3	1–2
Tensile strength	1	2–4	2–3	1–2
Material residue, toxicity	1	2	2–3	2–4
Induction period at 392°F. (200°C.)	3	2–3	1	2–3
Creep behavior	1	1	1	1–3

1 very good 4 questionable
2 good 5 bad
3 satisfactory

Table 8:1

The ratings refer to the property itself and/or to the backup data available for the particular property.

The Temperature Makes the Difference

The properties of the PEX materials depend upon the temperature at which the cross-linking process takes place. It is especially important to make a distinction between tubing that has been subjected to cross-linking at a temperature above the crystal melting temperature and one that has undergone the process below that temperature.

At temperatures above the crystal melting point (while the material is in an amorphous state), cross-linking occurs naturally without any chemical or mechanical disturbance. When cross-linking takes place below that temperature, it must occur among molecules in the amorphous area between crystallites and so the results are not as good. For example, the tubing that is developed in this way undergoes a change in some of its properties after hot bending. Being subjected temporarily to high temperatures has a great negative effect upon its strength due to the changes in the crystalline structure that it causes.

Tubing that undergoes cross-linking at temperatures below the crystalline melting point (for example, when using the silane or radiation cross-linking methods) normally loses from 10 to 15 percent of its strength when heated up to temperatures over that point. Tubing that is cross-linked using the Engel method does not share this loss of strength under similar treatment.

Creep

In the case of polymers as well as many other materials, the reponse to a constant load over an extended period of time is an elongation or deformation of a part or all of the material. This reaction, which increases over a period of time, is known as "creep". The deformation that occurs in such situations can be divided into an initial deformation and a creep deformation. The ratio of the initial deformation to the creep deformation is dependent upon the amount of the load or stress, the temperature of the material and the actual material being tested. (See Figure 8:1.)

Elongation (Deformation) is a Function of Time and Load (expressed as stress).

When comparing different materials it is important to:

1. Distinguish between the initial deformation and the creep deformation. (See figure 8:2.)
2. Make the comparisons between materials regarding creep relative to their strength (or elasticity modulus, stress rating, etc.). For example, the amount of creep for PVC at 2000 psi (25 N/mm^2) could be compared to the amount of creep for a specific PE at 400 psi (5 N/mm^2).
3. Conduct the comparison at temperatures commensurate with the materials being tested. For example, if the materials are to be used for hot water pipes, 180°F. (80°C.) would be an appropriate temperature.

The generalized curves shown in Figure 8:1 would be valid for three different temperatures (for example 70°, 140° and 230°F. or 20°, 60° and 110°C.) at the same stress as well as for three different stresses at the same temperature as is actually shown.

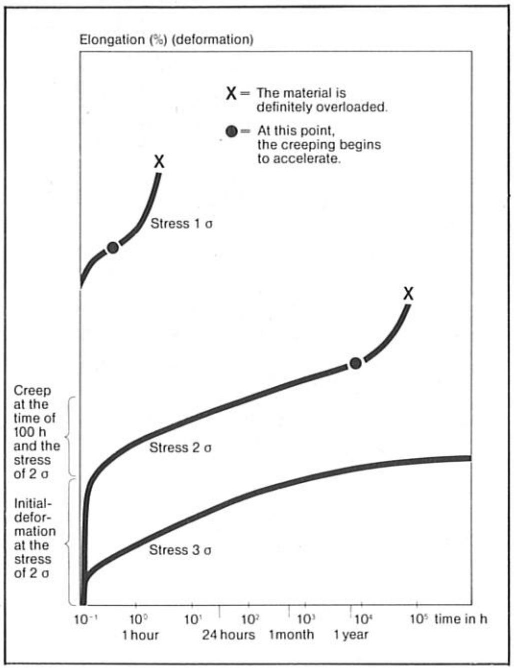


Figure 8:1

***At this point,** the creeping begins to accelerate. The material is definitely overloaded.*

***Creep Curves** Elongation (Deformation) is shown here as a function of time. Generalized creep curves at three different stresses (at a specific amount, at two times that amount and at three times that amount).*

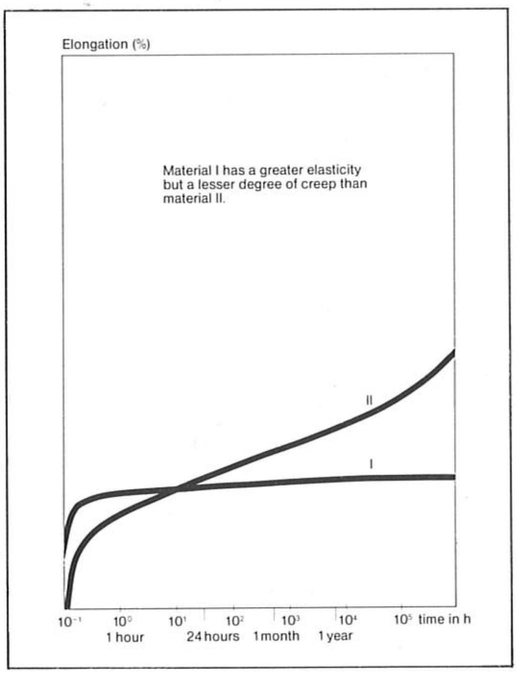


Figure 8:2

***Material I** has a greater elasticity but a lesser degree of creep than material II.*

***Creep curves.** This graph illustrates the difference between two materials. Material I undergoes a large initial deformation but a small creep. The opposite is true of material II. While its initial deformation is smaller, the degree of creep is larger.*