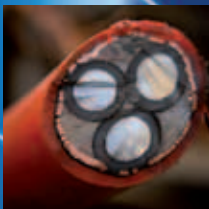


# Radiation Crosslinking

Enhancing Plastics Properties by Irradiation



**BGS**

IDEEN PLUS ENERGIE



*BGS: We optimize your products at three locations.*

## Radiation crosslinking by BGS

### What we do, we do with energy

Treating plastics with high-energy electron radiation or gamma radiation is the value-for-money alternative to expensive high-performance polymers. As a pioneer in this field, BGS Beta-Gamma-Service GmbH & Co. KG has more than 25 years' experience in the industrial application of beta and gamma radiation. We are much in demand as a partner in many different sectors, wherever there are new challenges to be mastered.

Radiation crosslinking gives inexpensive commodity plastics and technical plastics the thermal, mechanical and chemical properties of high-performance plastics. And it does so without affecting the production process: radiation crosslinking takes place after moulding, as the last step in the process chain – on the way to the final customer. The products can be delivered for irradiation as continuous products wound onto drums, loose in mesh boxes or – for injection moulded products – as bulk goods packed in cartons.

Radiation crosslinking is characterized by high process reliability and reproducibility, and saves plastics processing firms from having to make substantial investments. With many years of know-how and state-of-the-art radiation plants, BGS provides the optimal service for every customer. We help you to upgrade and extend the application of your plastic products and hence increase their value added, for instance by improving their thermal stability and their abrasion resistance.

When optimizing your products by high-energy radiation, rely on an innovative enterprise with certified quality: BGS.

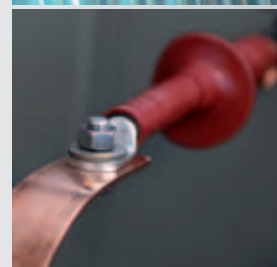
### BGS – your partner with many years' experience in the special treatment of sensitive goods

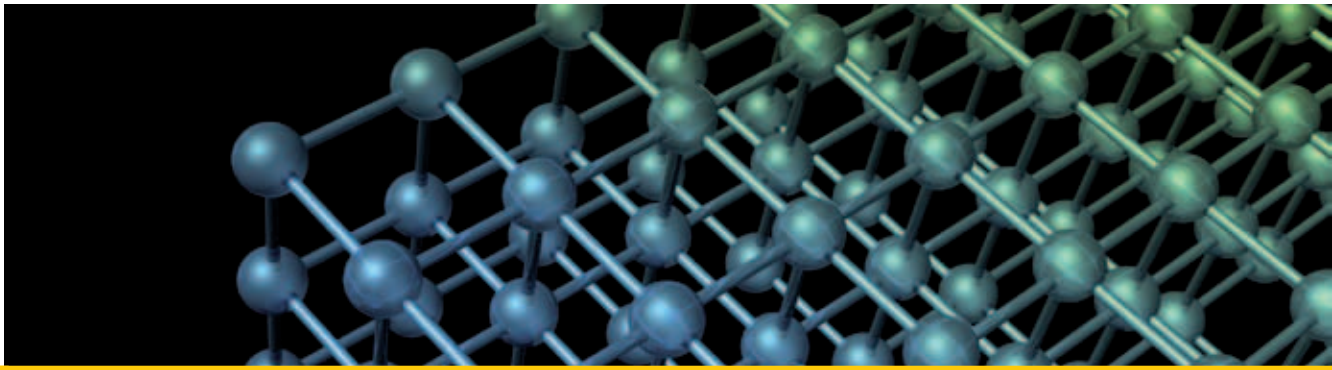
The comprehensive service of BGS expresses our wish to offer you a 'total care' package – whatever special tasks are involved:

- We integrate our services into your production process in a way which saves costs and resources: you send us the goods for irradiation, and they go on from BGS to their final destination.
- We support you already at the development stage, to ensure that your products are ideally adapted for subsequent radiation treatment.
- Our documented quality gives you certified safety.

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## Radiation Crosslinking of Materials

### The principle

Radiation crosslinking is based on the effect of high-energy beta and gamma rays. Polymers are exposed to an exactly predetermined dose of gamma or beta radiation and in this way the crosslinking of the molecules is accurately controlled. The required material properties are precisely defined beforehand, and then obtained by applying exactly the right irradiation dose.

In the process of crosslinking the material absorbs radiation energy. Chemical bonds are broken and free radicals are formed which, in the next step, react to new chemical bonds. Thus an extremely resistant 'network' is formed. Since it is the finished plastic product which is modified in this way, it is even possible to vary the degree of crosslinking within one component by shielding parts of the product during irradiation. The irradiation of raw materials is also possible.

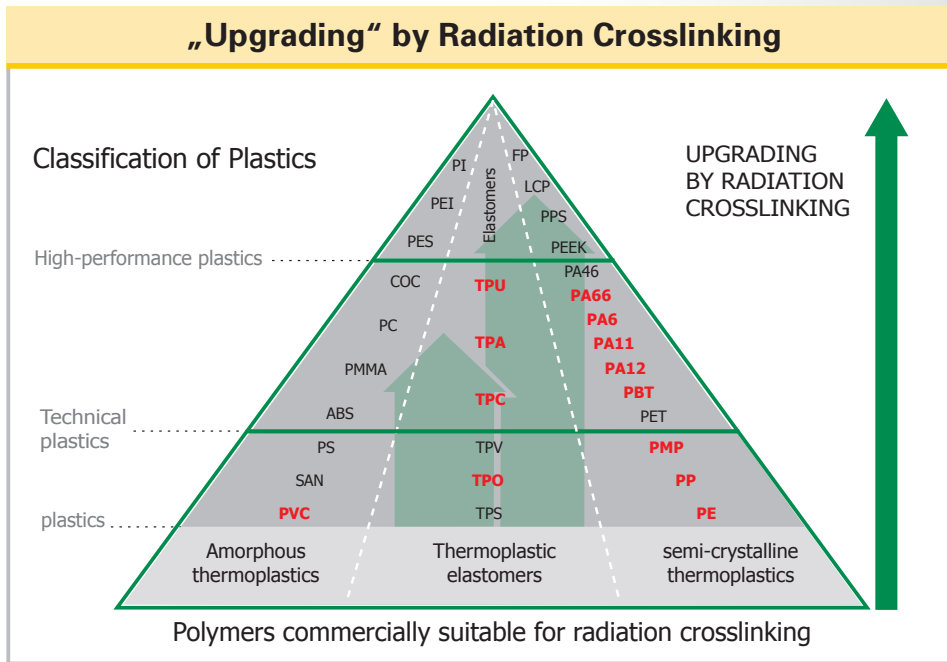
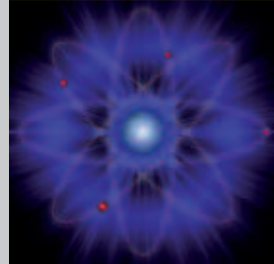
Radiation crosslinking is basically suitable for all types of plastics which can be chemically crosslinked by the use of radical initiators (e. g. peroxides). However, unlike chemical crosslinking methods, radiation crosslinking takes place at low temperatures. From the point of view of quantities, the most important polymers to be radiation crosslinked are polyethylene (PE), polyamide (PA), polybutylene terephthalate (PBT) and polyvinyl chloride (PVC). The crosslinking of thermoplastic elastomers (TPE) is becoming increasingly important and polypropylene (PP) can also be radiation crosslinked.

The way radiation crosslinking works can be compared with the vulcanization of rubber, a method that has been in industrial use for so long. As a physical process, irradiation has the advantage that the effects are obtained at low temperatures, and that results can be achieved with precision and without fluctuations in quality. By adjusting the use of beta or gamma rays to meet the specific requirements in each case, BGS helps to optimize the properties of plastics and opens up new fields of application for established raw materials.

Because crosslinking takes place as an external step after the manufacturer's production process, optimal process speed is not affected. Another advantage of irradiating the finished injection moulded parts is that plastic production waste (for instance mould gate residues) can simply be returned to the production process.

### Benefits of Radiation Crosslinking

- Savings in raw materials, as expensive high-performance polymers are no longer required
- New properties – and thus new applications – for certain raw materials
- Exactly reproducible processes
- Fast process
- Production waste is minimized



### From mass-produced to high-performance

“Upgrading” for plastics: radiation crosslinking gives commodity or technical plastics the mechanical, thermal and chemical properties of high-performance plastics. After undergoing radiation crosslinking, plastics can be used in conditions which they could not withstand otherwise.

Basically, radiation crosslinking takes place after the moulding process – injection moulding, extrusion or blow moulding is performed by the manufacturer as usual, with the established raw materials. This means that no costs for procuring new tools or machines are involved. Radiation crosslinking takes place at room temperature. It is a physical process in which radicals trigger crosslinking reactions in the polymers.

The spectrum of properties obtained depends on the basic polymer used. For some plastics, crosslinking activators are necessary. Crosslinking agents can be added during the production of the granulate (compounding) or directly before moulding (e. g. in masterbatches). The additives enable or improve crosslinkability and can further optimize the property profile of the plastic. The crosslinking agents used are well known from the plastic and rubber industry.

In order to evaluate the property changes that have been achieved, BGS can carry out special plastic tests after irradiation.

Crosslinkable Polymers			
Designation	Crosslinking additive		
	yes	no	
Thermoplastics:	Polyethylene PE (LLDPE/LDPE/MDPE/HDPE/UHMWPE)	x	
	Polypropylene PP (homopolymers/copolymers)	x	
	Polyamide PA (Polyamide 6/6.6/11/12)	x	
	Polybutylene terephthalate PBT	x	
	Polyvinylidene fluoride PVDF	x	
	Ethylene-tetrafluorethylene ETFE	x	
	Polyvinyl chloride PVC (only plasticised PVC)	x	
	Ethylene vinyl acetate (EVA)		x
	Chlorinated polyethylene (CPE)		x
Thermoplastic elastomers:	Polyether-ester block copolymer (TPE-E)		x
	Polyurethane block copolymer (TPE-U)	x	
	Polyether block amide (TPE-A)		x
Elastomers:	Styrene butadiene rubber (SBR)	x	
	Silicone rubber		x

## Changes in Properties

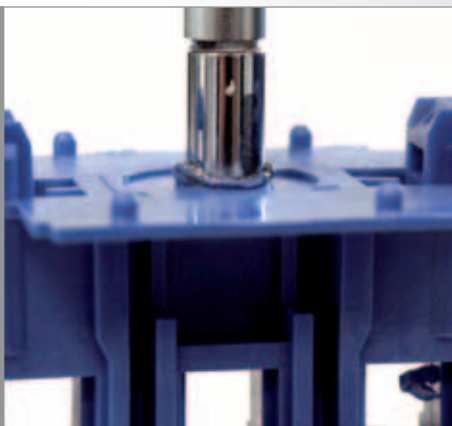
### ■ Improvements in thermal properties

When they undergo radiation crosslinking, thermoplastic materials become thermoelastic. The crosslinking reaction forms a network which inhibits the flow tendency of the plastic – at higher temperatures the material behaves like an elastomer. Improved thermal resistance and greatly enhanced mechanical values at high temperatures are characteristic features of radiation crosslinking.

For instance, with non-crosslinked PA-6.6 the moduli practically drop to zero above the crystallite melting temperature. By contrast, the substantially higher moduli of a crosslinked polymer give sufficient strength even at temperatures over 350 °C. Moreover, the thermal expansion coefficient is reduced. Crosslinked PA-6.6 shows a temperature index improvement of 20 °C (5,000 h; 60 % reduction of elongation at break).

Improvements in thermal properties resulting from irradiation:

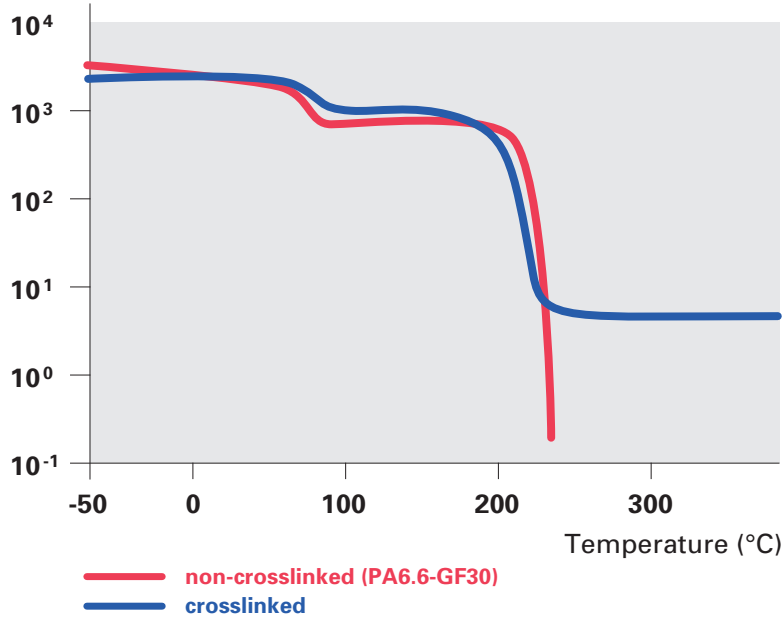
- Improved heat resistance with specifically-adjusted thermal expansion (Hot-Set/Hot-Modulus)
- Improvement in tensile set and compression set
- Higher hot wire resistance
- Higher ageing resistance



*Testing the heat resistance of crosslinked components using the soldering iron test (left: non-crosslinked, right: crosslinked, material: PA-6 GF 30, weight: 1,000 g, temperature: 350 °C).*

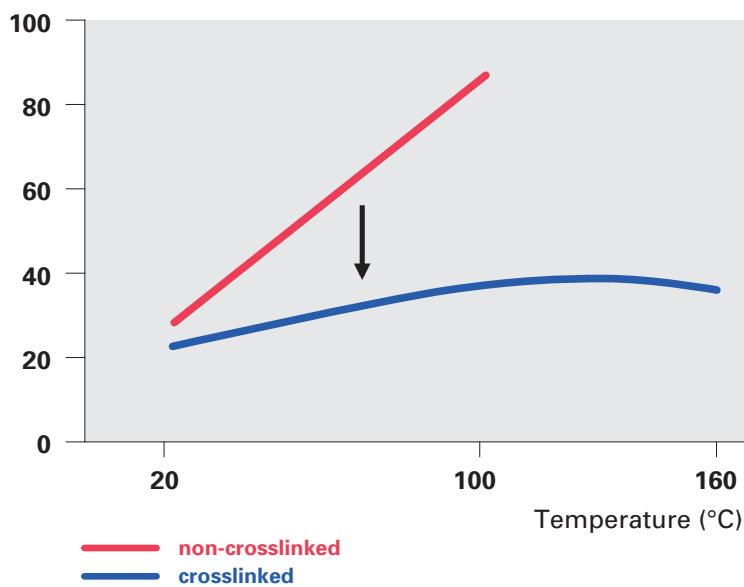
### Heat resistance of radiation crosslinked PA-6.6

Torsion module  $G'$   
(N/mm<sup>2</sup>)



### Compression set of radiation crosslinked TPE

Compression set (%)



The advantages of a crosslinked thermoplastic elastomer emerge clearly at high temperatures. Even at 160 °C, crosslinkable TPE compounds still have good compression set values, meaning that large portions of their property profiles are comparable to classic elastomers. Thus they are ideally suited for use as seals for parts made by 2-component injection moulding. Provided suitable materials are used for the casing, radiation crosslinking following production greatly improves the properties of both the injected TPE and the moulded part.



## Changes in Properties

### ■ Improvements in mechanical properties

Radiation crosslinking improves the mechanical strength of reinforced plastics even at room temperature. This is mainly due to better bonding of the fillers to the polymer matrix, caused by activation of the interfaces.

The weld line strength of vibration welded components and the bond strength between combinations of different materials (e. g. polymer/polymer and polymer/metal) are also improved by radiation crosslinking.

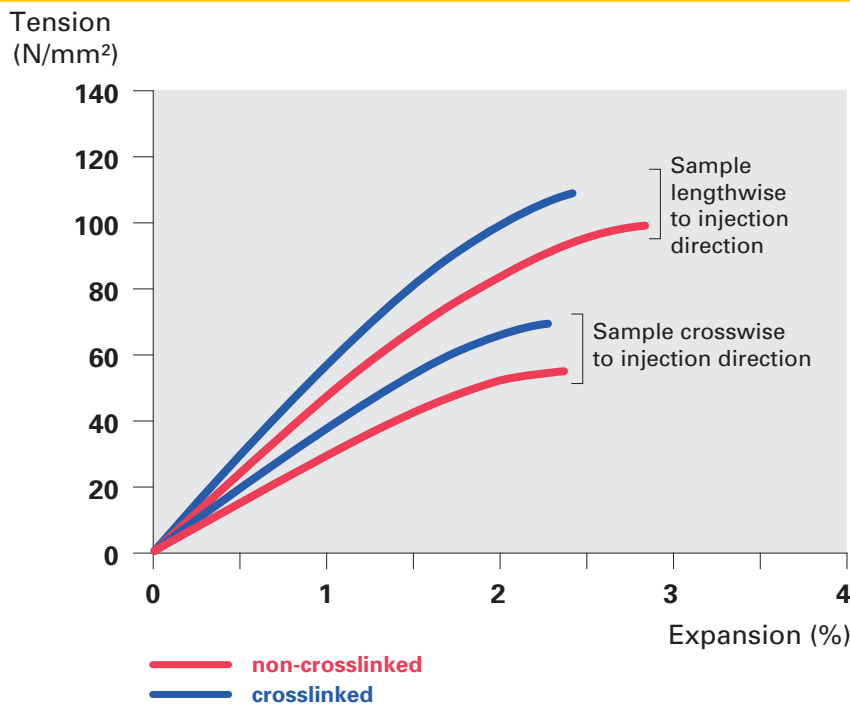
Improvements in mechanical properties resulting from irradiation:

- Increased moduli
- Reduction of cold flow (creep)
- Improvement in flexural strength (alternate bending strength)
- Increased weld line strength
- Improvement in long-term hydrostatic pressure resistance





### Increase in strength of PA-6 GF 30

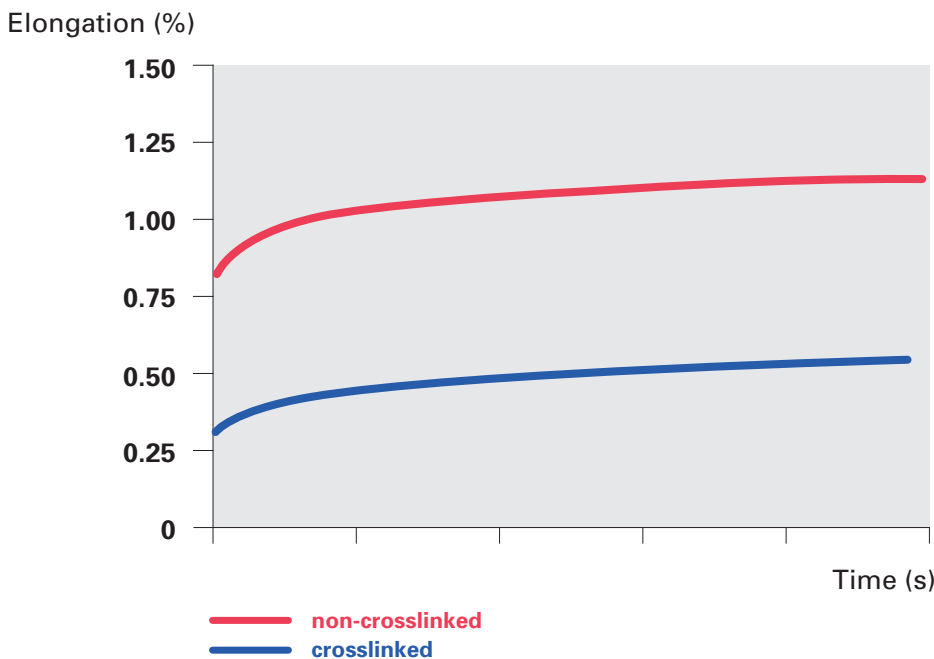


Improvement in fibre-to-matrix adhesion and strength in PA-6 GF 30 (top: non-crosslinked, bottom: crosslinked).



Particularly in glass fibre reinforced polyamide, irradiation produces substantial improvements in mechanical behaviour. Already at room temperature there is better adhesion between the glass fibres and the polymer matrix, and this makes the material much stronger. The reason: 'cross-border' crosslinking occurs at the interface between the glass fibre sizing and the plastic matrix.

### Creep behaviour of PA-6 GF 30 at 100 °C and 15 N/mm<sup>2</sup>



When plastics are subjected to mechanical load they tend to creep. The prestressing force, and thus the functionality of the plastics, is lost. Radiation crosslinking reduces the creep tendency of polymers. This is clearly shown in the creep curve for crosslinked PA-6 GF 30.



## Changes in Properties

### ■ Improvements in tribological properties

One important criterion when selecting plastic elements for machines is their friction and wear behaviour. The working temperatures that sliding bearings and gearwheels have to withstand are getting higher all the time, and friction and wear shorten their service life. Generally the relatively high proportion of amorphous areas at the surface of the plastic components (due to the production process) exhibit poor wear behaviour. These amorphous areas are particularly responsive to radiation crosslinking, which dramatically improves their wear behaviour. For instance, radiation crosslinking can raise the continuous working temperature of polyamides by up to 100 °C and can prevent melting. Crosslinked parts made of polyamide-6.6 have considerably higher sliding speeds, combined with reduced wear coefficients.

Tribological properties improved by irradiation:

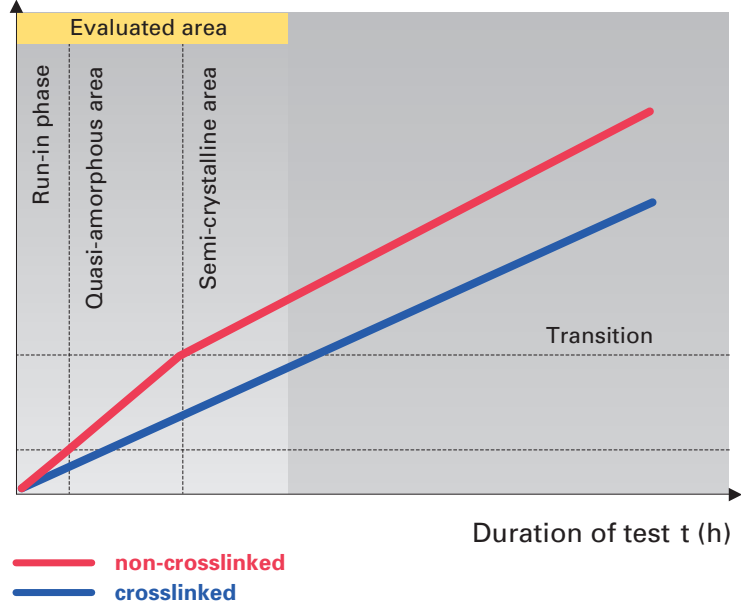
- Higher wear resistance
- Greater resistance to frictional heat damage: no melting of the sliding surface
- Higher dimensional stability
- Versatile lubrication options
- Higher mechanical strength (transferred momentum / gearwheels)





### Linear abrasion of PA-6.6 samples as a function of test duration

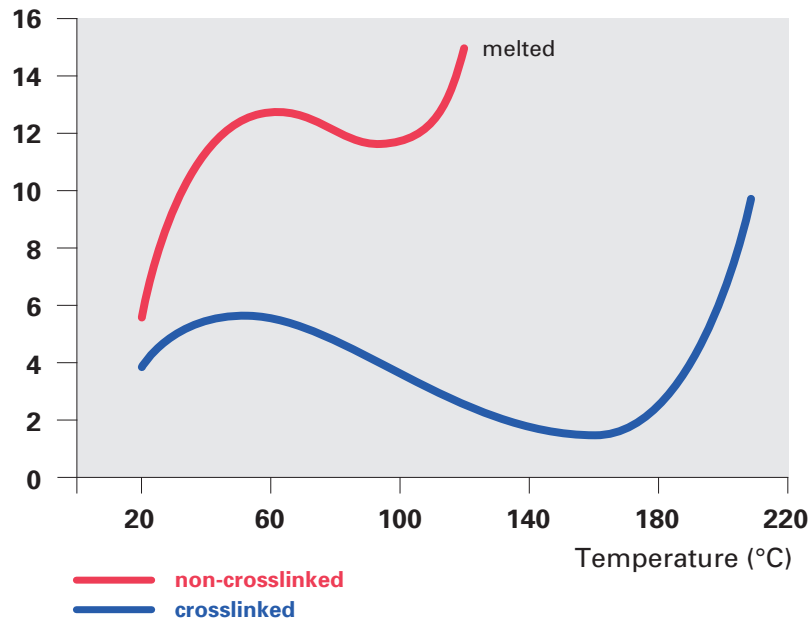
Linear abrasion ( $\mu\text{m}$ )



Crosslinked and non-crosslinked polyamides differ markedly in their abrasion behaviour. Crosslinked polyamides show an even level of abrasion over the whole duration of the test, and there is less abrasion altogether. The quasi-amorphous surface areas of the non-crosslinked polyamides show less resistance and wear faster than the semi-crystalline areas.

### Influence of ambient temperature on the wear coefficient of PA-6.6

Wear coefficient ( $10^{-6} \text{ mm}^3 / \text{Nm}$ )



The maximum operating temperature for non-crosslinked polyamide-6.6 under tribological stress is around  $120^\circ\text{C}$ . Radiation crosslinking inhibits melting and raises the maximum operating temperature by as much as  $100^\circ\text{C}$ , while at the same time reducing the wear coefficient. The higher thermal resistance means that the minimum rate of wear is only reached at about  $170^\circ\text{C}$ .



## Changes in Properties

### ■ Improvements in chemical properties

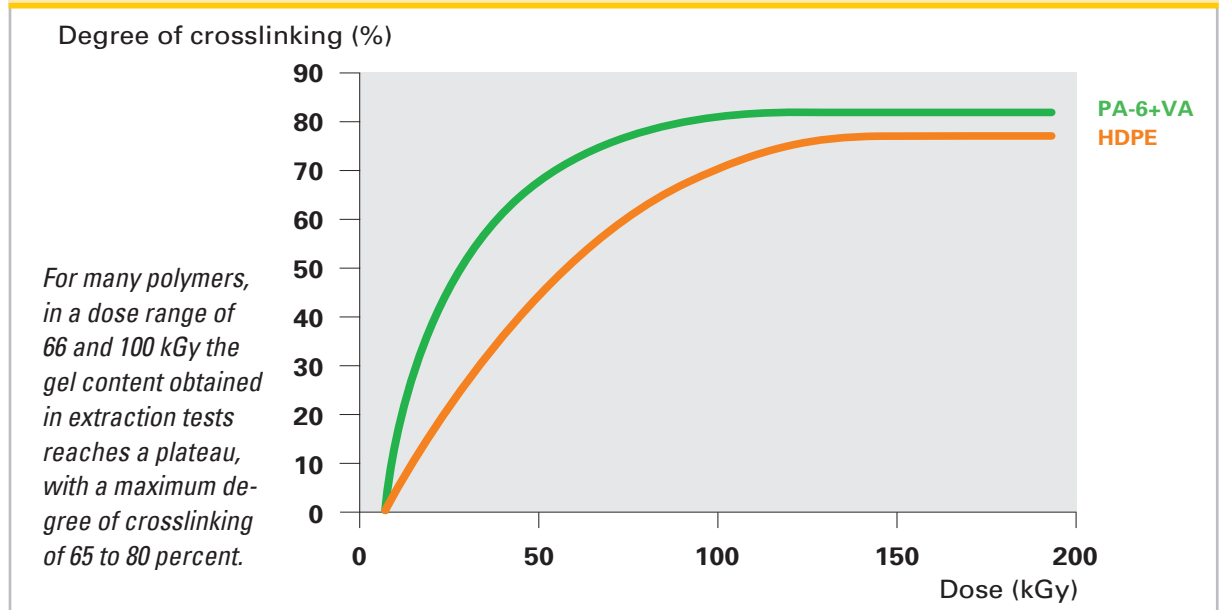
Crosslinking of plastics substantially reduces their solubility and swelling in solvents. This fact is used, for instance, when determining the degree of crosslinking by extraction tests. The 'gel value' obtained in this way correlates in a wide range with the degree of crosslinking: as the degree of crosslinking increases, the degree of swelling decreases.

Similarly, radiation crosslinking improves resistance to aggressive substances (e. g. brake fluid) and to hydrolysis. This is shown, for example, by improved resistance to stress cracking and a substantially reduced loss of strength after exposure to solvents.

Improvements in chemical properties resulting from irradiation:

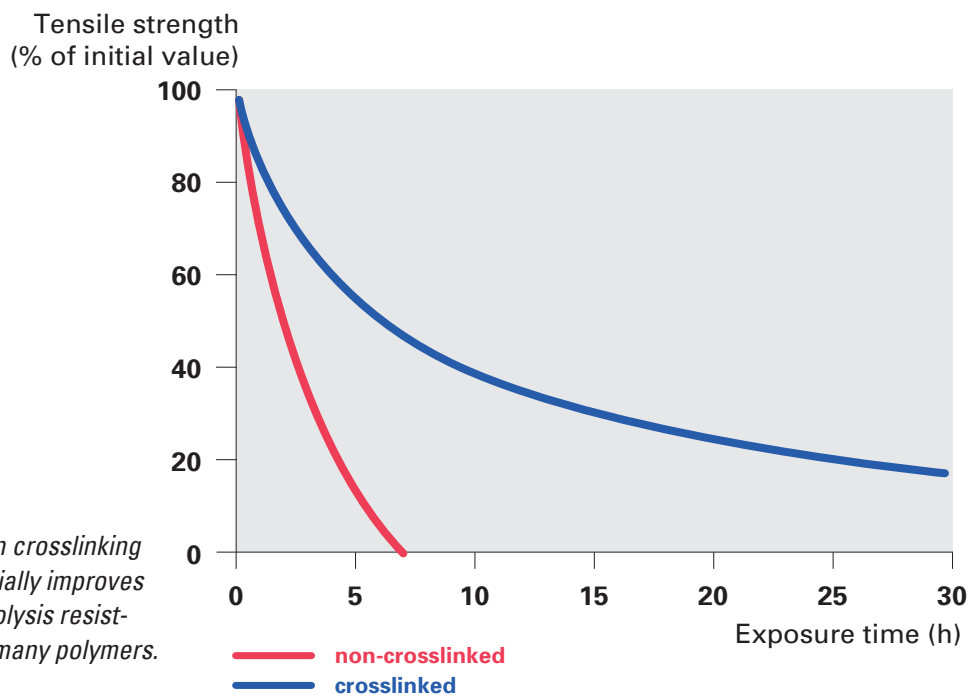
- Reduced solubility
- Improvement in swelling behaviour
- Greater resistance to stress cracking
- Improved resistance to hydrolysis and oil

### Gel content (degree of crosslinking) as a function of dosage





### Hydrolysis resistance of TPE-U in boiling water



### Resistance of PA to stress cracking



Crosslinking markedly improves resistance to stress cracking, here in PA-6. After submersion in a 30 % solution ( $ZnCl_2$ ) the non-crosslinked sample (left) shows clear evidence of stress cracking, whereas the crosslinked sample (right) shows virtually none.



## Applications

### ■ Electrical applications

In the electrical industry plastics are used for their good insulating properties and almost limitless shaping possibilities. Progressive miniaturization and new production technologies are continually making higher demands in terms of thermal stability and non-flammability. Thus, for instance, the lead-free solders commonly used today generate peak temperatures over 250 °C, which normally can only be withstood by high-performance plastics such as LCP, PEEK, PES or PEI. However, in some cases these are very difficult to metallize.

Radiation crosslinking of PA or PBT considerably extends the short-term upper temperatures to which components can be exposed without softening or melting. At the same time the bond strength between the metallization and the plastic surface is improved.

Today, the housings of sophisticated functional components for contactors are also made from radiation crosslinked PA instead of thermosetting plastics. If a short circuit occurs, the high temperatures in the arc no longer cause the plastic to melt and stick the contact surfaces together. Another positive effect of radiation crosslinking is the reduced flammability in the presence of hot wires (Hot Wire Ignition HWI according to UL-746A). This means that it is possible to abandon the use of flame retardants and still meet the requirements of the Standard UL-508.

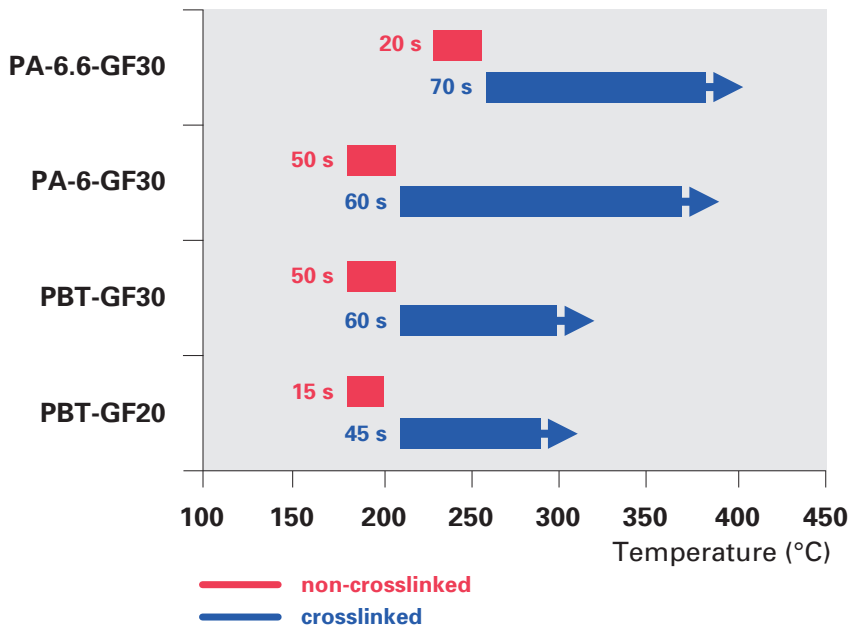
Improvements to chemical properties resulting from irradiation:

- Reduction of solubility
- Improvement of swelling behaviour
- Increased resistance to stress cracking
- Improvement in resistance to hydrolysis and oil



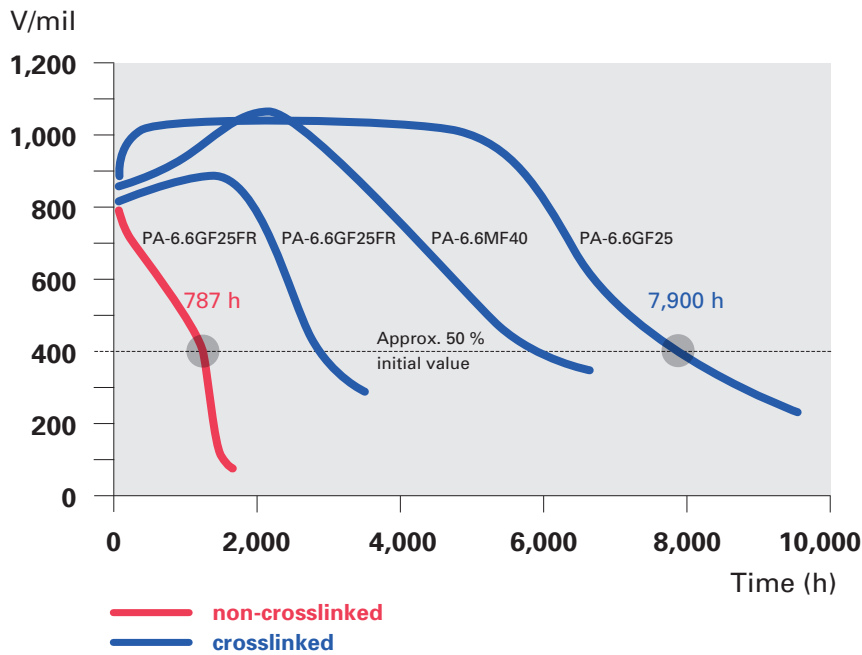


### Extended thermal limits for short-term immersion in a molten metal bath



Radiation crosslinking results in a marked improvement in duration and temperature limits for immersion in a molten metal bath.

### Ageing behaviour (temperature index RTI) of PA-6.6



The test describes the ageing behaviour of a plastic after long exposure to a high temperature. After ca. 800 hours, non-crosslinked PA-6.6 only retains 50 % of its electrical insulating properties. The life of a radiation crosslinked polyamide is improved by a factor of 10.



## Applications

### ■ Cables/wires

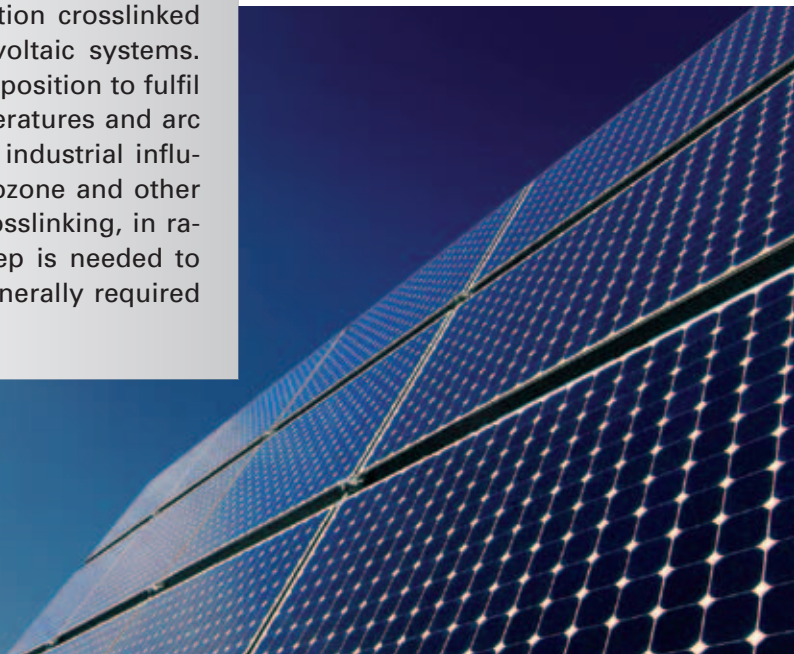
For years now, radiation crosslinking has ensured that plastics used to insulate cables and wires are able to fulfil the stringent requirements for resistance to heat and chemicals. Tried and tested applications include, for instance, improved resistance to welding beads in cables and leads in vehicles, in order to meet the heat resistance requirements classes C and D (working temperatures of up to 125 °C and 150 °C respectively). Today, energy cables are also successfully radiation crosslinked to obtain special properties.

Radiation crosslinking allows more flexibility in the choice of raw materials and in the design and construction of cables. Not only individually insulated cable cores but also several cores twisted together, or complete leads and cables, can be crosslinked in a single process step. If the application requires the use of an inner core insulation which is sensitive to radiation, it is even possible to crosslink the outer sheath only. If desired, BGS gives support in optimizing the radiation dose and the polymer compound used, in order to identify the best option in terms of technical properties and costs. These possibilities make radiation crosslinking a competitive alternative to other crosslinking processes.

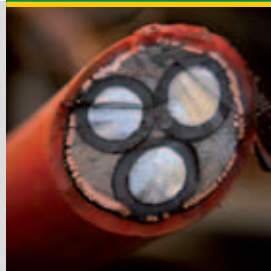
Another important application for radiation crosslinked cables are connecting cables for photovoltaic systems. Only radiation crosslinked cables are in a position to fulfil the legal requirements for working temperatures and arc resistance, and to withstand stress from industrial influences such as acid rain, exhaust gases, ozone and other chemical substances. Unlike chemical crosslinking, in radiation crosslinking only one process step is needed to crosslink the double insulating sheath generally required for photovoltaic cables.

Improvements in properties resulting from irradiation:

- Higher limit values for working temperatures
- Resistance to welding beads
- Increased resistance to heat pressure
- Better resistance to oil and chemicals
- Better resistance to hydrolysis
- Higher flexural strength (alternate bending strength)
- Better abrasion properties
- Improved resistance to stress cracking

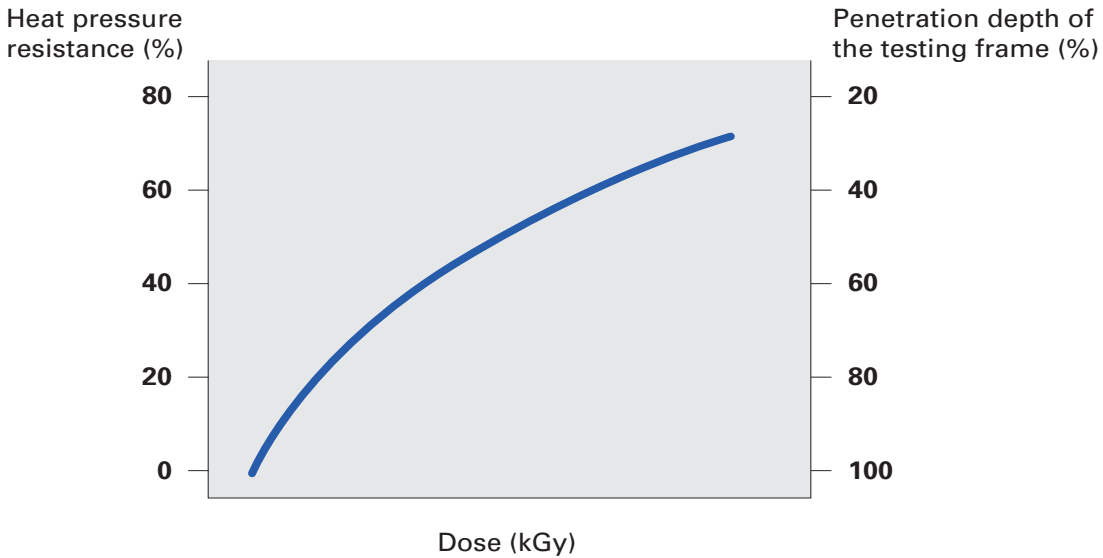






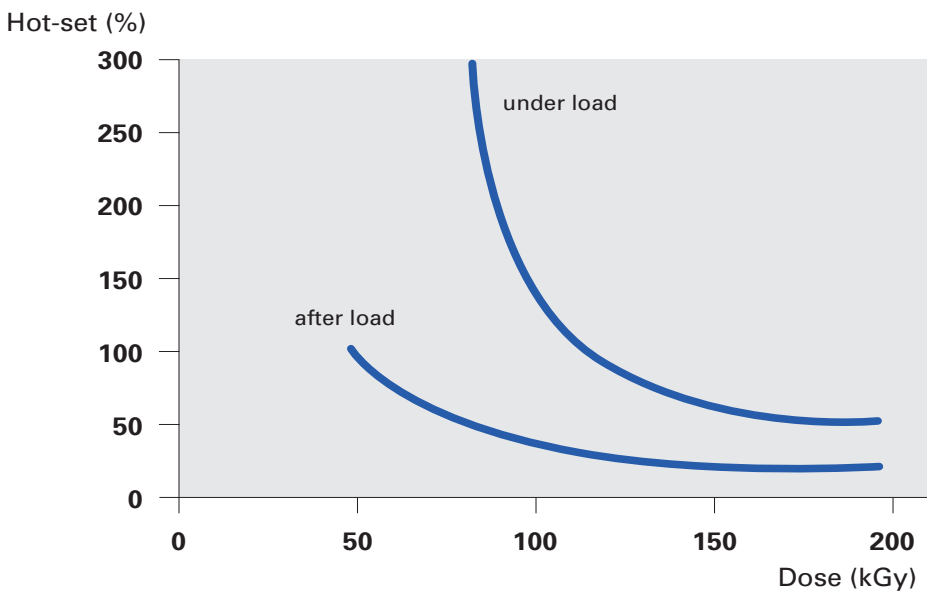
Compared with chemical methods, the physical process of radiation crosslinking offers very good process reliability. The process is precisely controllable and reproducible and there is no possibility of the fluctuations that may occur in chemical crosslinking. Another advantage of radiation crosslinking is its speed. It is much faster than other methods.

**Compressive strength of radiation crosslinked thermoplastic polyurethane (150 °C, 4 hrs.)**



*The thermal resistance of the cable sheaths can be greatly improved by radiation crosslinking.*

**Halogen-free, flame-retardant polyolefin-based cable compound (200 °C, 15 min., 20 N/cm<sup>2</sup>)**



*The desired expansion under a specific load at high temperatures (hot-set value) can be adjusted for the specific material by varying the radiation dose.*

*Radiation crosslinked automotive components fulfil the most stringent requirements regarding temperature and resistance to chemicals. They also have improved abrasion behaviour.*



## Applications

### ■ Automotive

Plastics are becoming continually more important as raw materials in the automotive industry, in order to meet the demand for reduced weight and lower fuel consumption. However, the materials are exposed to high thermal, mechanical and chemical stress if they are used, for instance, for applications in the engine compartment or exhaust system. Traditionally, this requirement profile can only be fulfilled by using more expensive high-performance plastics, which are also more difficult to process. Here, radiation crosslinking offers a solution by extending the application range of less expensive materials that have already been introduced (e. g. PA). This helps to save raw material costs and also makes it possible to limit the growing numbers of raw materials.

If the plastics are additionally subjected to high temperatures, aggressive substances or high mechanical loads, radiation crosslinking improves their ageing behaviour. Even thermoplastic elastomers – including polyester elastomers, whose use is often limited by insufficient compression sets – can be so effectively optimized by radiation crosslinking that they fulfil the stringent requirements of automotive engineering. Other possible applications are, for instance, elastomer seals, which are now manufactured by two-component injection moulding and then radiation crosslinked. Provided a suitable combination of materials is used, the housing and the injected TPE seal can be crosslinked in a single process step.

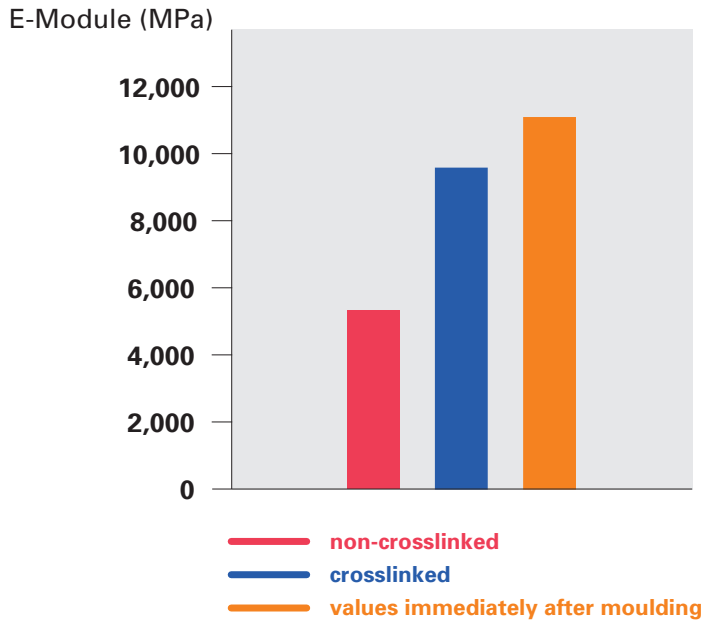
Improvements in properties resulting from irradiation:

- Improved strength and creep resistance
- Higher heat resistance and lower thermal expansion
- Improved ageing resistance
- Reduction of swelling and better resistance to stress cracking
- Improved compression set
- Improved abrasion values and a lower frictional coefficient
- Higher burst pressure
- Improved weld line strength

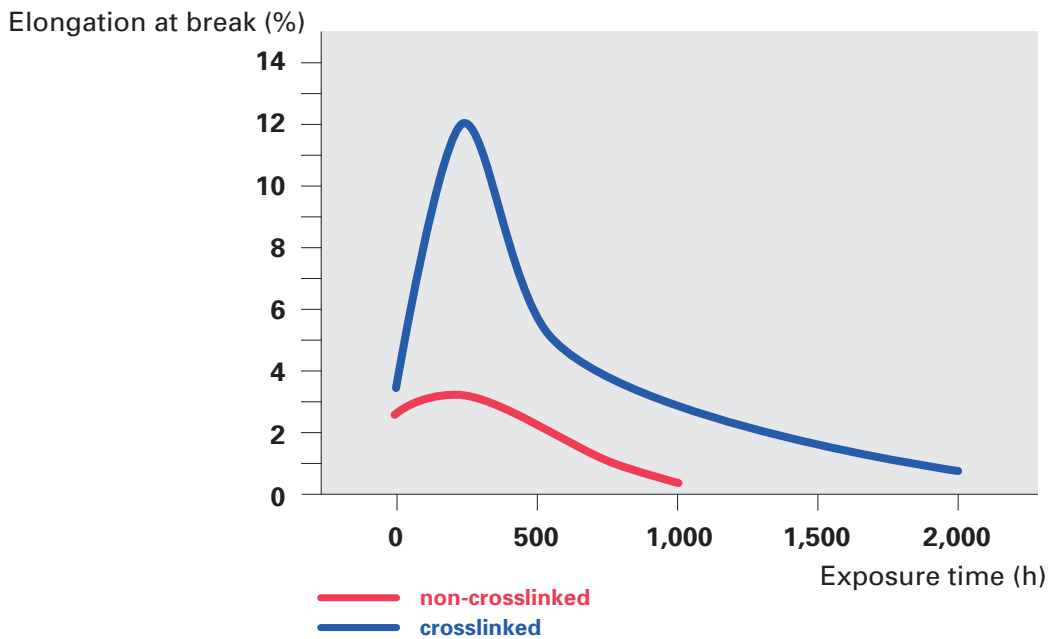




### Long-term ageing at 200 °C over 1,000 hours, PA-6.6 GF 30



### Submersion in coolant at 130 °C (glycol/water 50:50) of PA-6.6 GF 30



*In automotive engineering, parts come into contact with high temperatures and aggressive substances. This limits the use of many materials. Here, radiation crosslinking greatly improves long-term durability.*



*The most usual material for the manufacture of pipes is PE. However, media-conveying tubes and pipes which have to satisfy the highest requirements in terms of temperature resistance and burst resistance are made from radiation crosslinked PA-11.*

## Applications

### ■ Pipes/tubes

For decades now, plastic pipes made from HDPE have been radiation crosslinked in order to ensure that they keep their performance characteristics for a very long time. Of particular importance is their improved durability with respect to high temperatures and internal pressure. For more than 30 years, several million kilometres of radiation crosslinked pipes – known as PE-Xc pipes – installed all over the world have demonstrated their reliability in day-to-day use under difficult conditions. Unlike chemically crosslinked pipes, which are designated as PE-Xa and PE-Xb, with radiation crosslinked PE-Xc pipes there is no risk of residues from crosslinking chemicals. Moreover, the physical process of radiation crosslinking offers very high process reliability and substantially higher production speeds than chemical crosslinking processes.

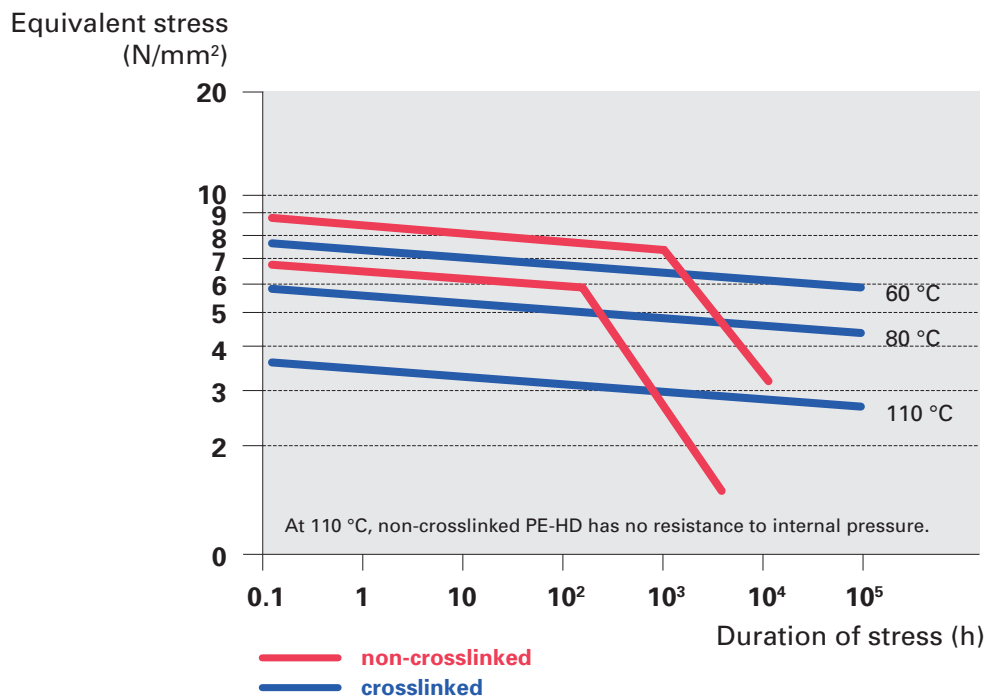
Improvements in properties resulting from irradiation:

- Higher durability over time at high temperatures
- Improvement in cold flow values
- Improved resistance to chemicals
- Reduced crack propagation
- Pressure resistance
- Crosslinking of the outer and inner layers can take place in one process step, even with metal composite pipes
- All colours crosslinkable as desired
- Improved flexural strength (alternate bending strength)
- Better resistance to soldering beads



*Radiation crosslinked corrugated pipes have demonstrably better durability characteristics and better resistance to high temperatures. This is important, for instance, in their use as protective cable conduits.*

## Long-term durability of HDPE under internal pressure



Applications:  
pipes/tubes



Continuous products on drums:

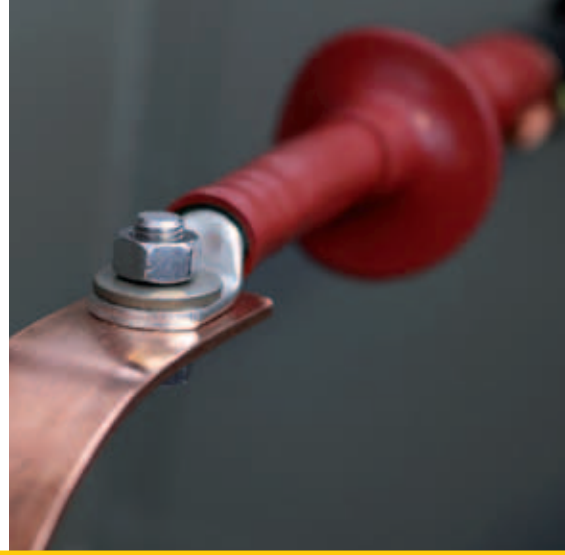
- Metal composite and PE pipes
- Pipe diameter: 5 to 90 mm
- Max. drum diameter: 3.20 m
- Max. drum width: 2.60 m



Product lengths:

- Metal composite and PE pipes
- Max. length: 12 m
- Max. pipe diameter: 600 mm
- Max. wall thickness: 20 mm

*Heat shrink products are used in electrical insulation, and as seals for cables and pipelines. They owe their 'shape memory' to radiation crosslinking.*



## Applications

### ■ Heat shrink products

Heat shrink technology is an important area of application for radiation crosslinked polyolefins, for instance in the electrical industry and in pipeline construction. Typical 'shrinkable' products are tubes, foils and moulded parts. In these heat shrink products, semi-crystalline materials are given a 'shape memory' by the selective formation of crosslinking points. The shape memory is due to the fact that radiation-induced crosslinking occurs mainly in the amorphous areas. When a product that has been crosslinked in this way is stretched under heat, this shape can be temporarily 'frozen in' by cooling it below the crystallite melting temperature. When the user then heats the product up again above the crystallite melting temperature, it returns to the original shape at the time of crosslinking.



*The crosslinking sections created by radiation crosslinking are responsible for the restoring forces that are activated by warming. Heat shrink products are used in the electrical industry and for sealing cables and pipelines. Radiation crosslinking gives them their shape memory.*

## Other applications

In principle, the changes produced by irradiation such as crosslinking, branching (grafting) or molecular weight reduction can also be applied to polymer raw materials. Examples of such applications are a controlled increase of the molecular weight of ethylene (co)polymers, or introducing long-chain branching in order to obtain higher processing viscosities or melt strengths.

The deliberate degradation of polymer chains by irradiation is used with polypropylene. PP modified in this way works as a nucleating agent and speeds up the crystallization of non-reinforced PP, if added in small amounts of 2 to 3 percent.

Particularly with thick-walled moulded parts, cooling and cycle times can be greatly reduced by adding irradiated PP granulate. Moreover, a much finer semi-crystalline structure is obtained, resulting in other property improvements – for instance, higher heat resistance, increased stiffness and impact strength, and a reduction of creep.

PTFE can be degraded to powders by irradiation and is used as an additive to improve lubrication in various different technical applications.

### Improvements in properties resulting from irradiation:

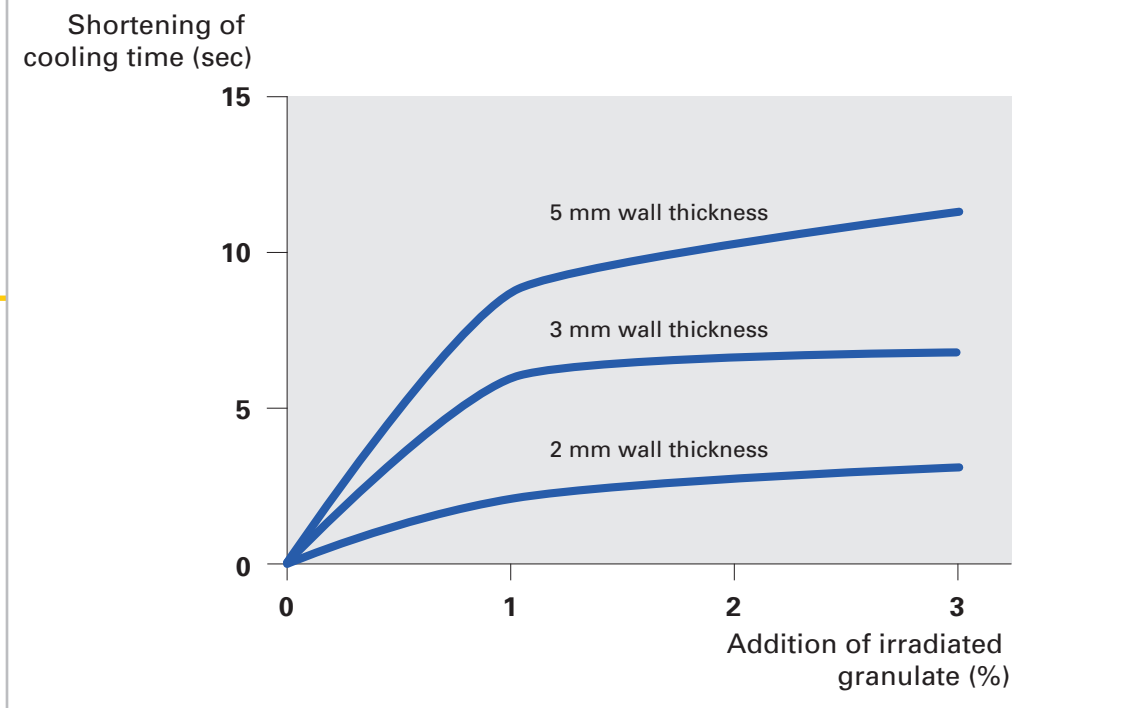
- Depending on the polymer structure, modification of the rheological properties (e. g. melt viscosity, melt strength)
- Better processability
- Production of PTFE powders
- Destruction of microorganisms (radiation sterilization)



*Irradiation reduces the molecular weight of the plastic, and as a result the mechanical properties decline. This situation can be creatively exploited, for instance in plastic ampoules made of PP: irradiation makes the predetermined breaking point so brittle that the neck can be broken off splinter-free.*



## Shortening of cooling time as a factor in the addition of irradiated PP-granulate



*The processing properties of many polymer raw materials can be optimized by irradiation. Depending on the molecular structure, new bonds are formed or existing bonds broken. In this way, new properties can be obtained and valuable additives produced.*







## Recycling



Radiation crosslinked parts are extremely rugged and can thus be used for a very long time. When one of the durable plastic parts has reached the end of its working life, there are – as for plastics in general – three recycling options: material utilization (physical), raw material utilization (chemical) or energetic (thermal) utilization.

Material reutilization results in new plastic components produced from the secondary raw materials. Provided production residues (such as sprues and rejects) are pure and sorted by type, they can simply be returned to production before crosslinking takes place. Material utilization also works if crosslinking additives are included (“regranulate”).

Crosslinked plastics in pure form can be shredded and remixed with primary raw materials in the form of regranulate within certain limits. The limits depend on the material and the degree of crosslinking, and have to be individually checked in each case.

If physical recycling is not possible or does not make sense, radiation crosslinked components can be chemically or energetically recycled without any problems.



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