A Practical Approach to Product Dosimetry in a Multi-site Company

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A Practical Approach to Product Dosimetry in a Multi-site Company

1 – Use reference dosimeters only where necessary.

2 – Use the relative physical, chemical or electrical changes induced into the product by irradiation as a means of indicating crosslinked density.

These changes are indicative of product performance.

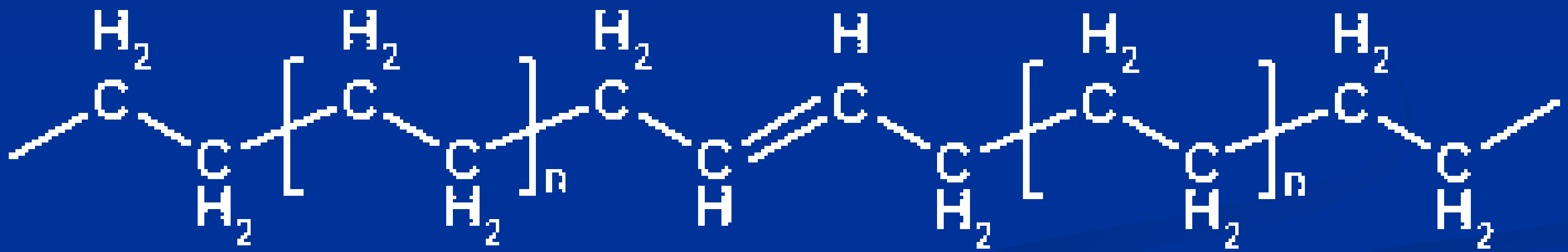
Polyethylene Dosimeter Properties – a reference dosimeter

+ Fundamental change in the structure of the polymer
  Development of the transvinylene double bond
    Observed by Dole and Charlesby (1950's)
    Elaborated on by Lyons and Johnson (1995 and 2004)

+ Detectable by Fourier Transformation Infra-red
  FTIR absorbance at 965 cm^{-1} wavenumber

+ Acknowledged ASTM standard in Medical Device area
  ASTM F-2381 Standard Test Method for Evaluating Trans-vinylene Yield in Irradiated UHMWPE

Double Bond Formation in Irradiated PE



Transvinylene double bond
in irradiated polyethylene

Polyethylene Dosimeter Properties – a reference dosimeter

+ Wide dynamic range (10 kGy to > 750 kGy)
+ Conformable
+ Thickness independent
+ Temperature independent
+ Humidity independent
+ Light insensitive
+ Stable and permanent record
+ NIST traceable secondary standard
– Dose-rate dependence at very low dose rates

Transvinylene in PE

"Transvinylene formation in polyethylene is substantially unaffected by such variables as temperature, dose rate, branching or degree of crystallinity, film thickness or presence of protecting additives."

Arthur Charlesby (1963)

Polyethylene Dosimeter Methodology

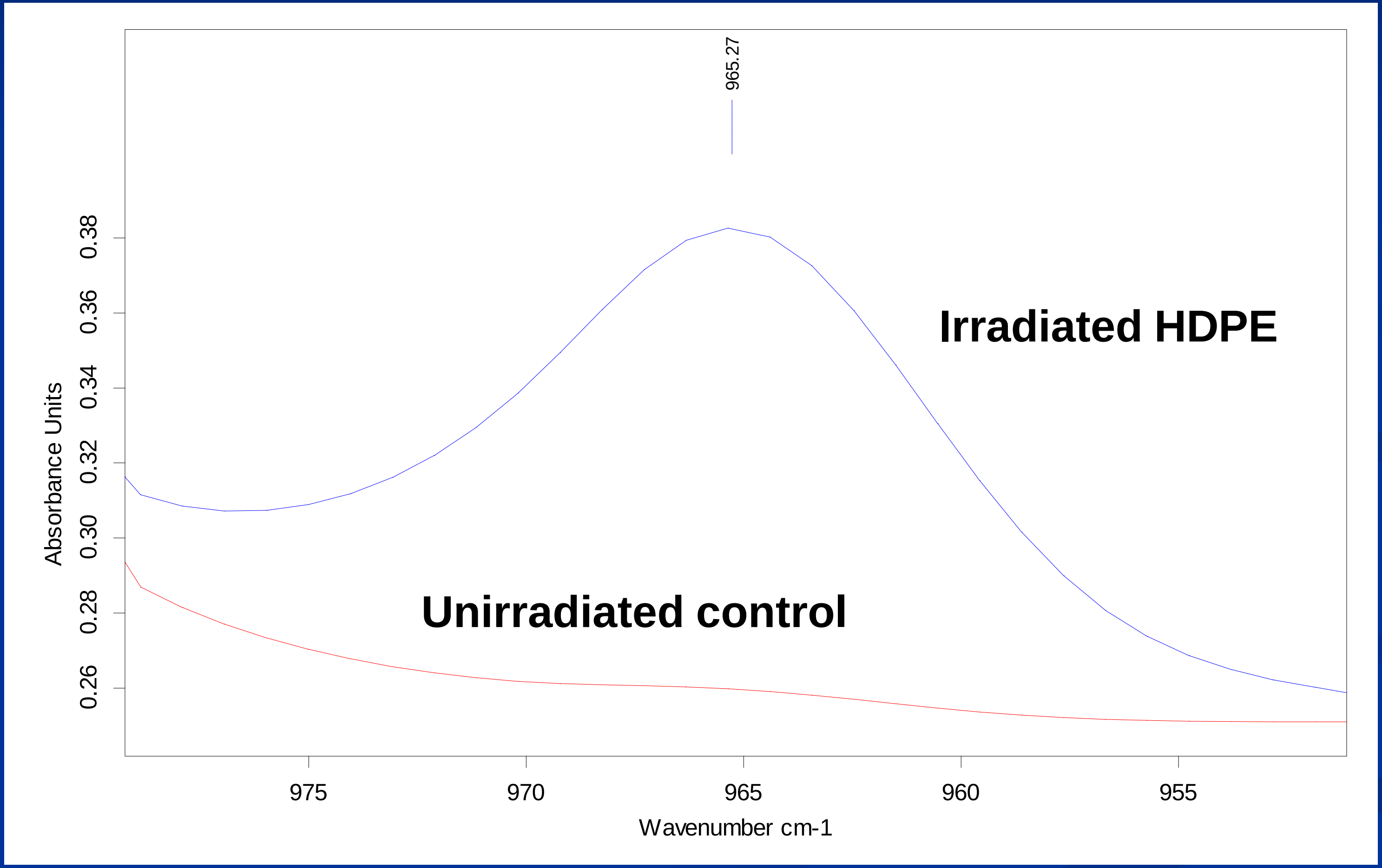
+ Extrude sheet material to have minimal orientation.

+ Stock sheet material can have different thickness (10 mils/254 µm, 20 mils/508 µm, etc.)

+ Growth of transvinylene absorption peak at 965 cm^{-1} is dose dependent.

+ Polyethylene absorption peaks at 1363 cm^{-1} or 2019 cm^{-1} can be used as references to eliminate any variations due to thickness.

Transmission FTIR Absorbance at 965 cm^{-1} in 890 μm HDPE Sheet



30 kGy at 3.0 MV

Use of Polyethylene Dosimeters in Research and Development and in Manufacturing

+ In research related to the radiation crosslinking of polymers and in the development of materials to be used in the manufacture of crosslinked products, the determination of dose is necessary.

+ Different polymers, different PE types and grades have different responses to irradiation. The irradiation response of a polymer should be determined before it is used in a formulation.

Research and Development Activities

+ Investigation of potential crosslinkable polymers:
  by literature studies and by comparison to other polymers in the same class

+ Product development:
  dose needed to attain the required properties
  determination of dose uniformity

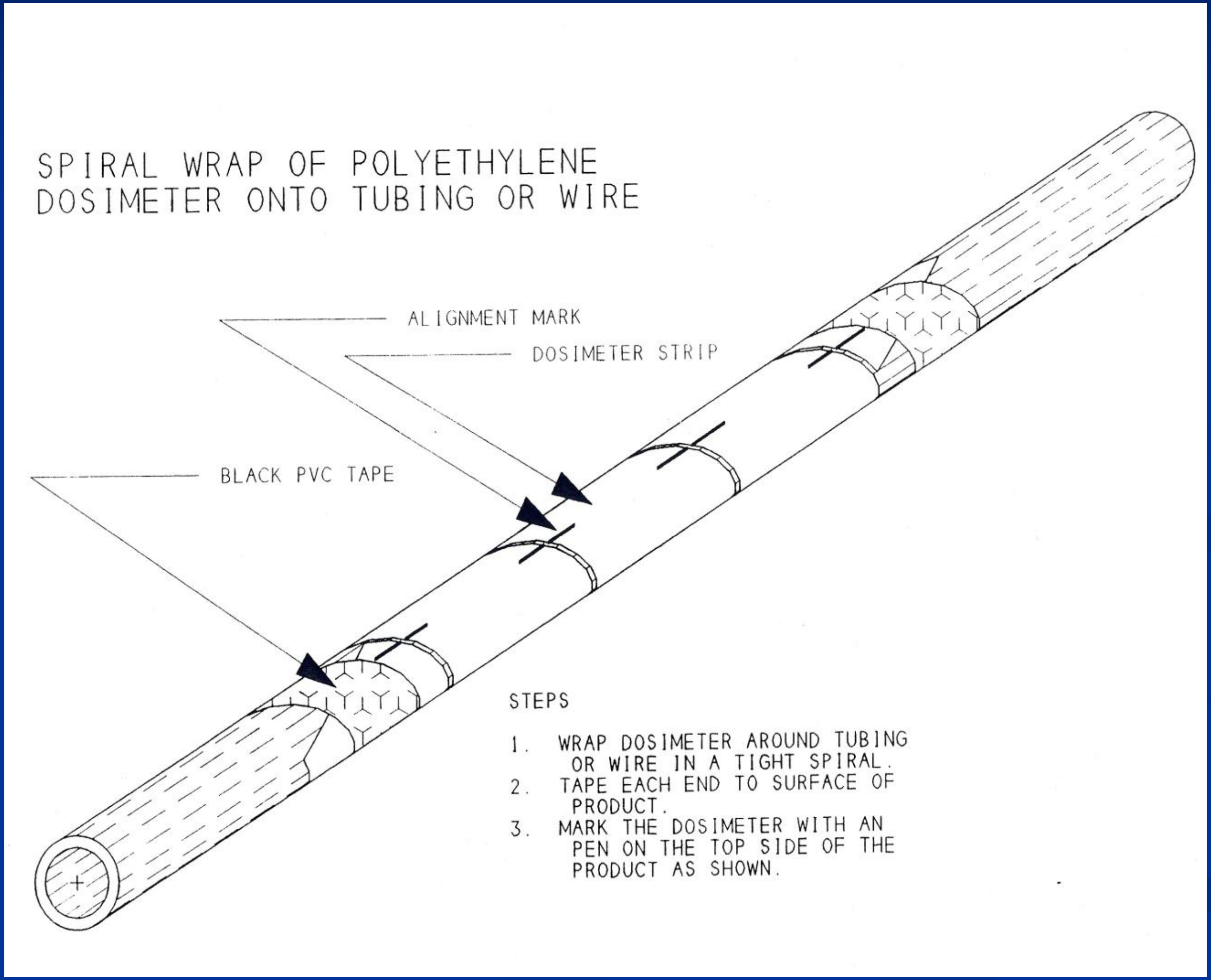
+ Inter-comparisons between company R&D centers

+ Writing publications and patents, doing presentations

Use of Polyethylene Dosimeters in Manufacturing

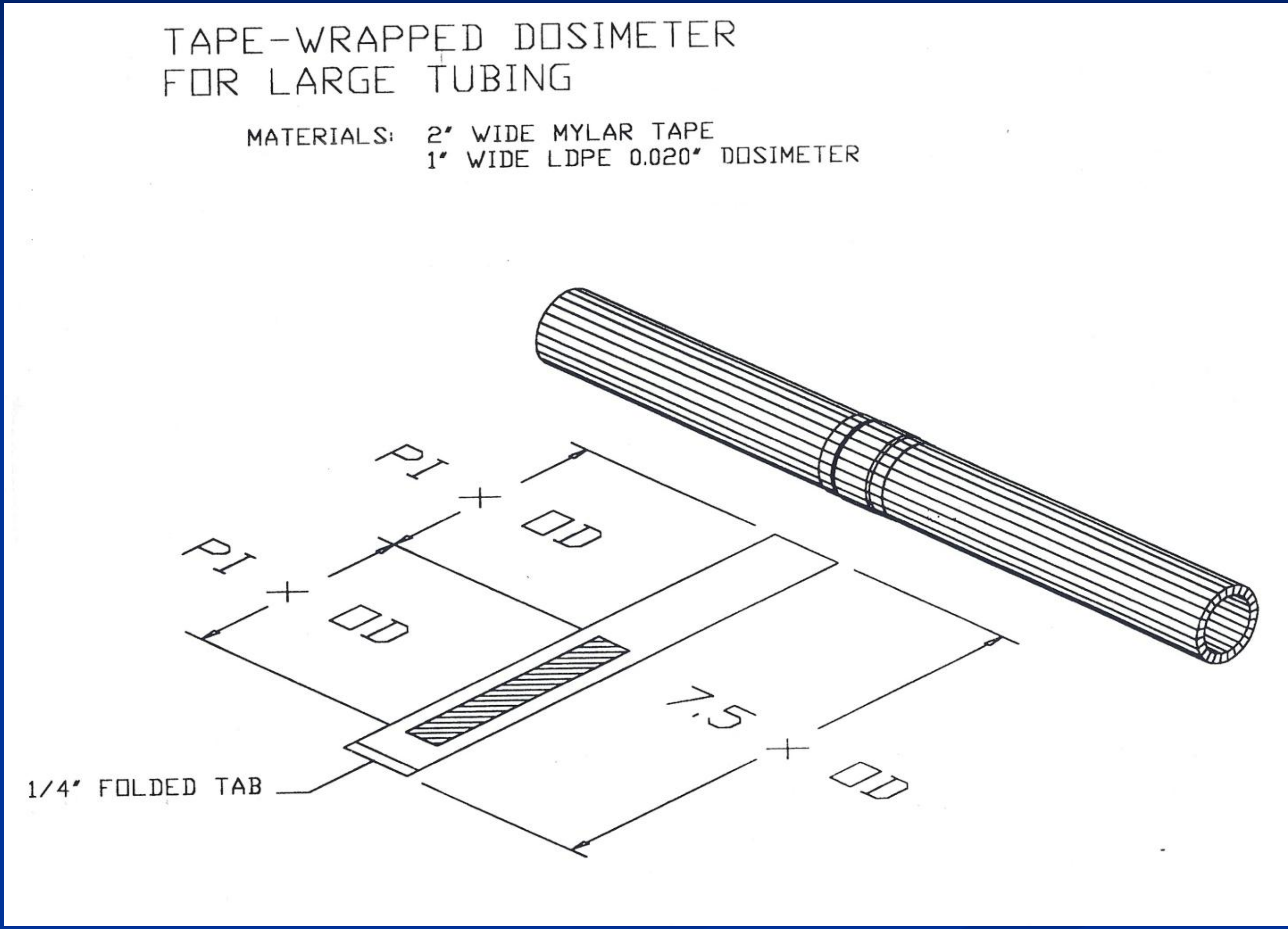
+ Production plant floor quality control
+ Inter-plant comparisons
+ Customer contractual requirements
+ Dose uniformity measurement
+ Product and process trouble shooting
+ Radiation equipment calibration and trouble shooting

Dose Uniformity Measurements



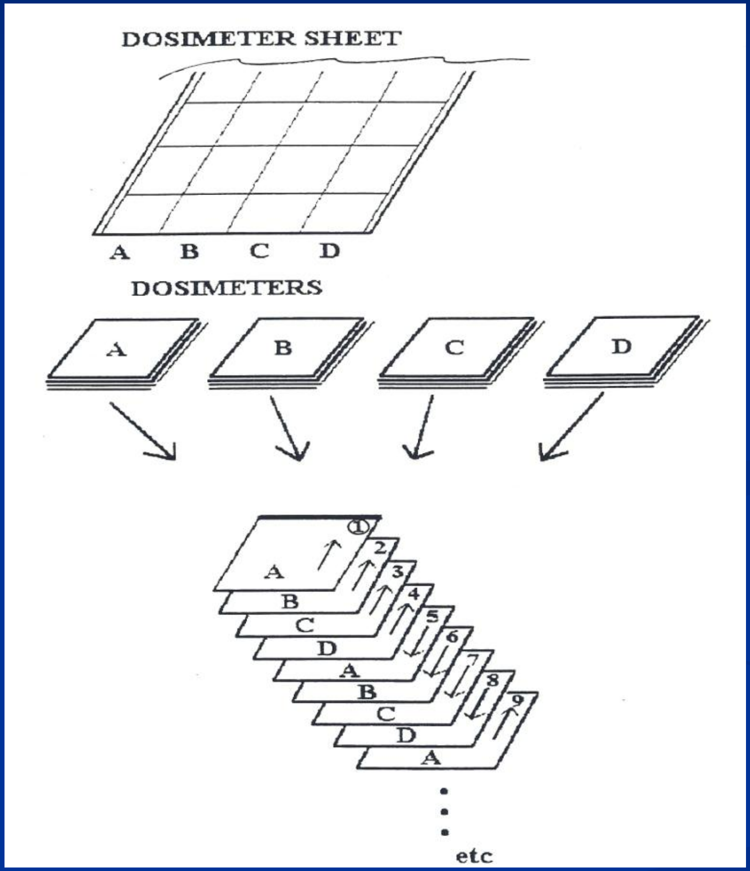
Small tubing or wire dosimetry

Dose Uniformity Measurements



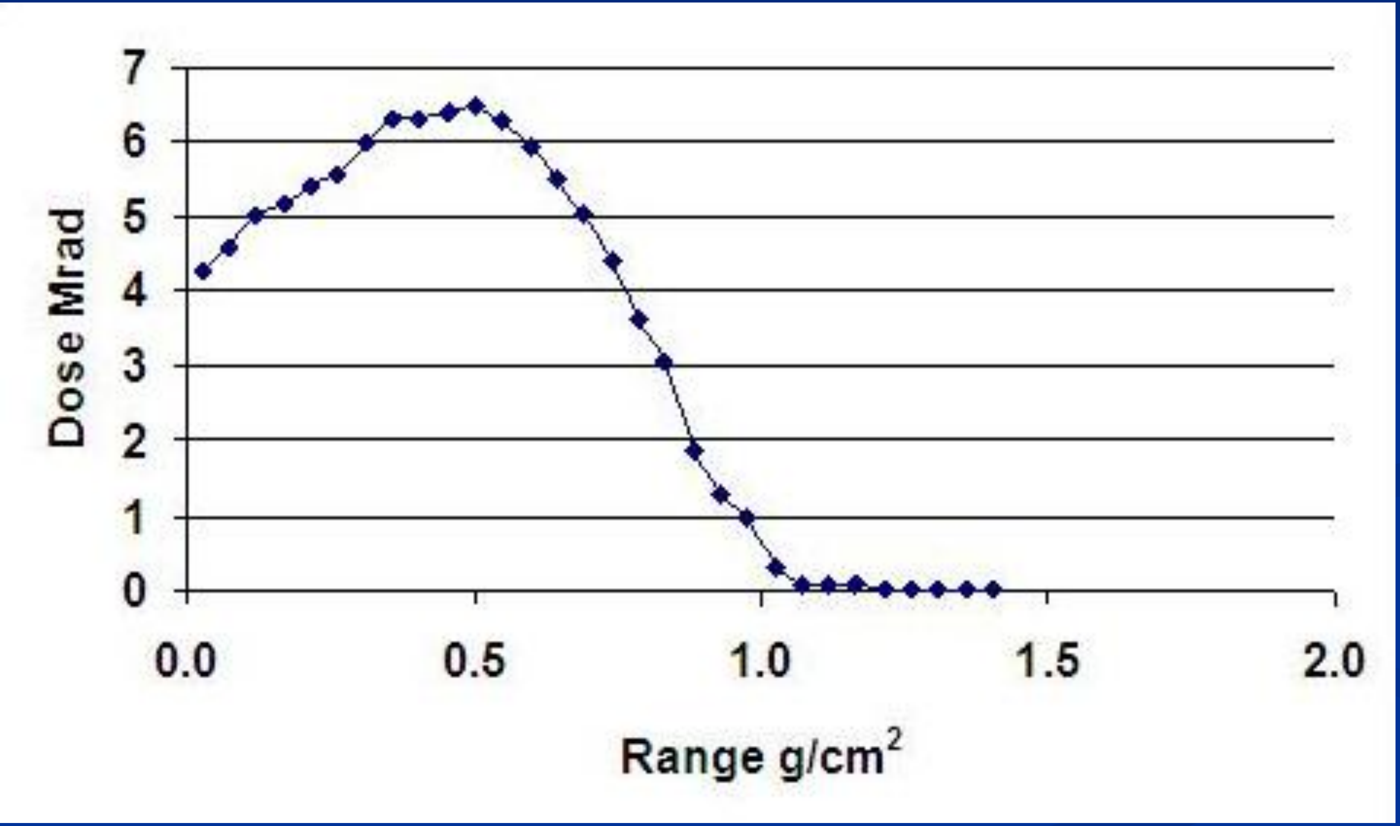
Large tubing cable dose uniformity

Equipment Calibration: Electron Beam Energy



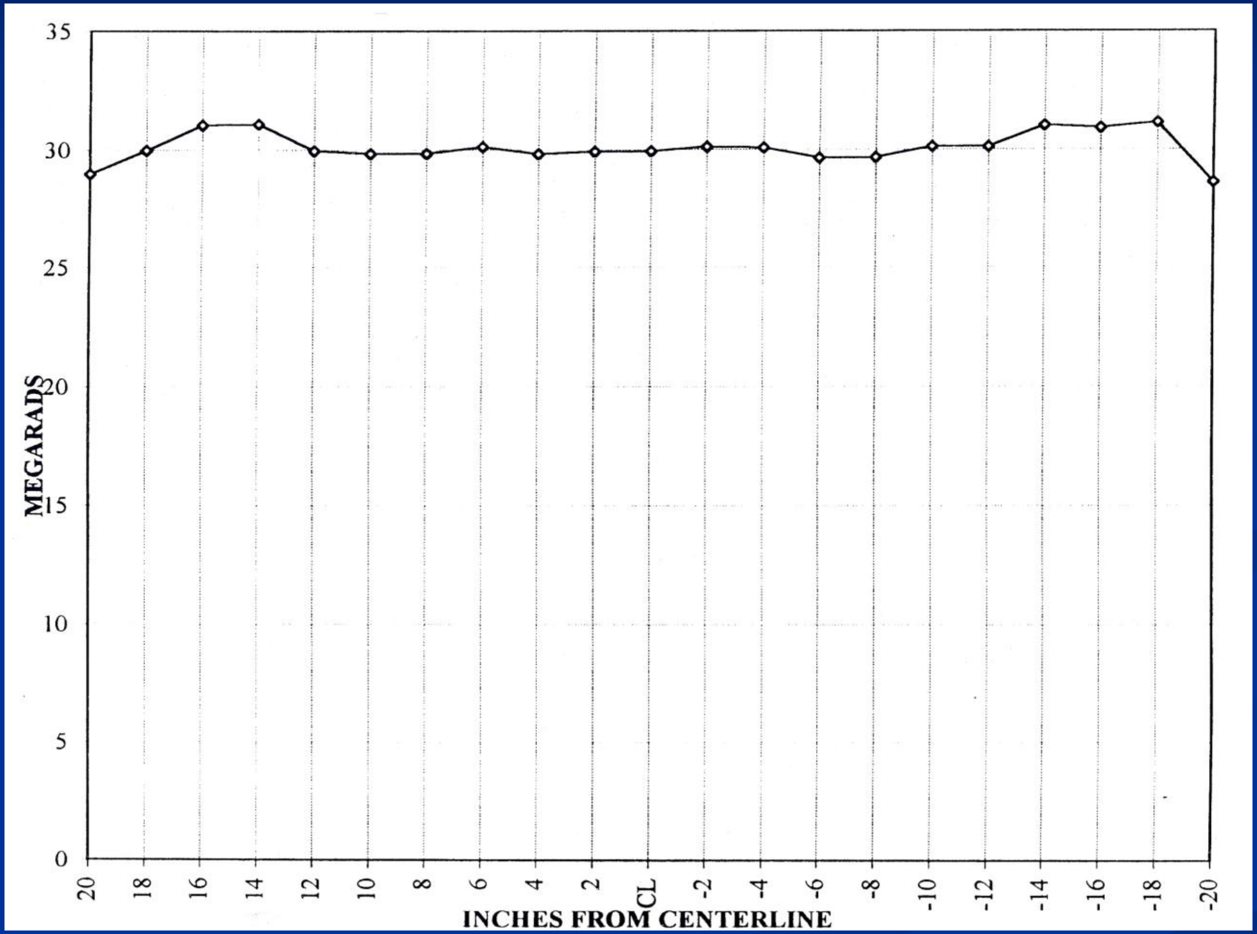
Construction of a depth-dose pack using polyethylene sheet material

Equipment Calibration: Electron Beam Energy



Depth-dose curve obtained using
a polyethylene depth-dose pack

Equipment Calibration: Electron Beam Scan Pattern



Electron beam scan pattern obtained using a strip of polyethylene dosimeter material

Manufacturing Processes

- To obtain consistent and reliable irradiated products such as wire, heat recoverable tubing, polymeric self-limiting heaters and Polyswitch® devices, all materials and manufacturing processes must be well defined and controlled.

Manufacturing Processes

+ In-coming raw material specifications and in-plant quality assurance inspections

+ Compounding
  - complex materials
  - time before forming (compatibility)
  - equipment operation

+ Forming (extrusion, molding)
  - blending
  - orientation
  - time before irradiation (compatibility)
  - time after irradiation (free radical lifetime)

The Irradiation Process

+ Electron beam characteristics
  - penetration depth – product density
  - spatial distribution – scan uniformity

+ Under beam handling equipment
  - speed
  - tension control
  - dose uniformity – wire, shaped products

Irradiation Processing in Manufacturing

+ Functional Dosimetry – based on and correlated with physical, chemical and electrical changes in irradiation processed materials

+ Physical, chemical and/or electrical changes in irradiation processed materials meet product and customer specifications

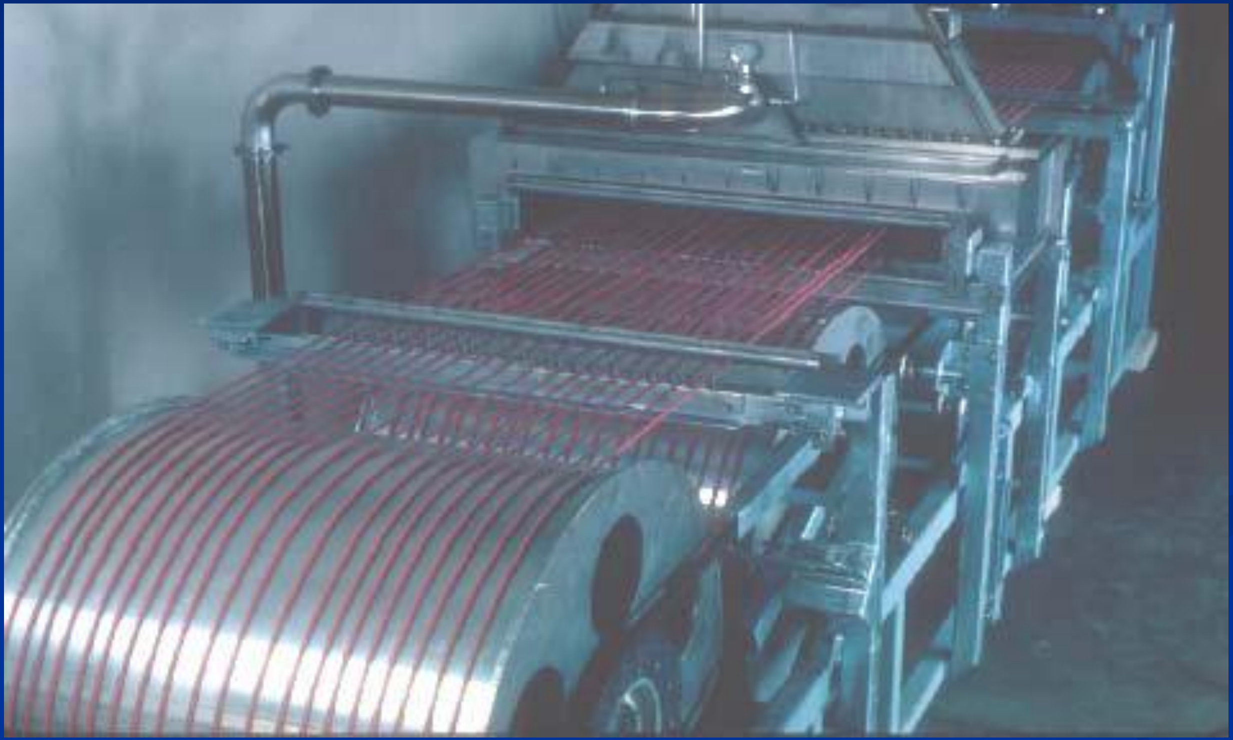
Property Changes Induced by Radiation Crosslinking

- + Increased hot modulus (modulus of the formulated polymer while in the melt)
- + Improved aging characteristics
- + Improved cut-through and abrasion resistance
- + Improved chemical resistance
- + Improved flame resistance – retention of incompatible additives
- + Changes in electrical properties – conductivity versus temperature
- + Changes in optical properties – relative changes in the infrared absorption spectrum

Property Changes Used for Product Dosimetry and Quality Control

+ Hot modulus (modulus above melt temperature) – production plant floor
+ Per cent shrinkage above melt temperature – production plant floor
+ Elongation to break – production plant floor
+ Heat aging – testing laboratory
+ Melt resistance test (solder iron test)
+ Cold bend – testing laboratory
+ Resistance versus temperature – testing laboratory

Product Dosimetry
Wire and Cable

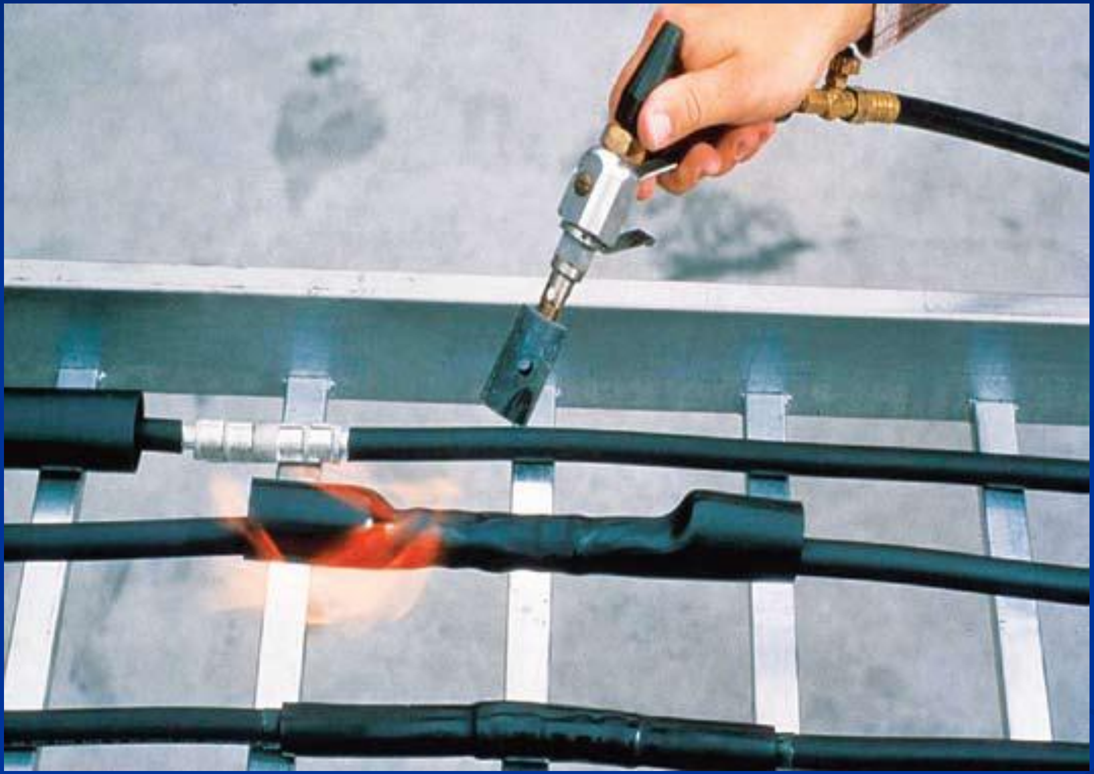


Tests: Tensile strength and elongation to break
Cold bend test – insulation cracking
Flammability testing and heat aging

Product Dosimetry
Wire and Cable

Flammability test: flame retarded irradiation crosslinked wire jacketing does not drip or fall away from the conductor

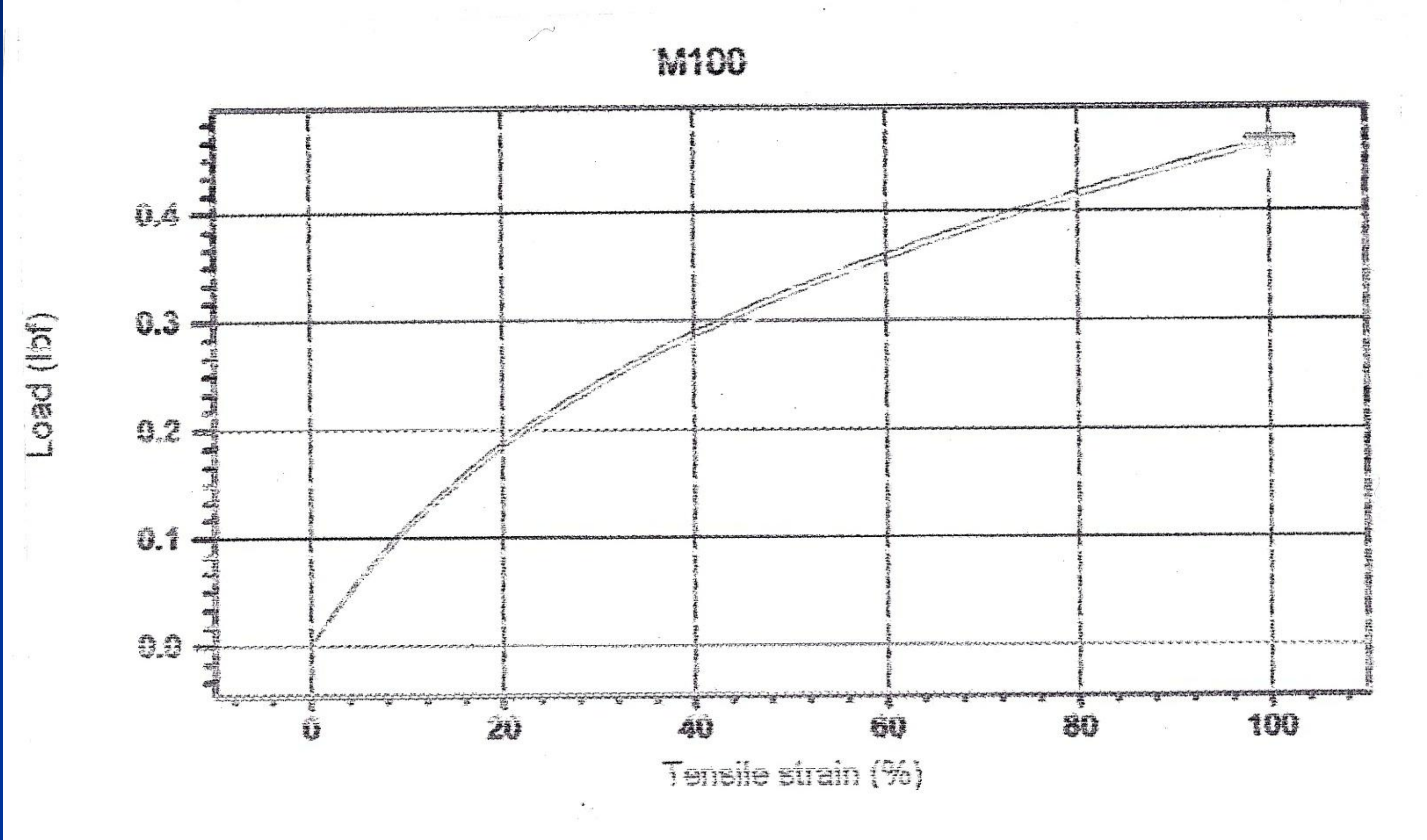
Product Dosimetry
Heat Shrinkable Tubing



Hot modulus (M-100) = modulus at 150°C, 100% elongation

Per cent shrinkage/recovery

Hot Modulus Testing



Modulus at 150°C determined using an tensile tester

Controlled Hot Modulus Heat Shrinkable Tubing

Too high a hot modulus = difficulty expanding the tubing
= possible spitting on recovery
Too low a hot modulus = insufficient recovery force

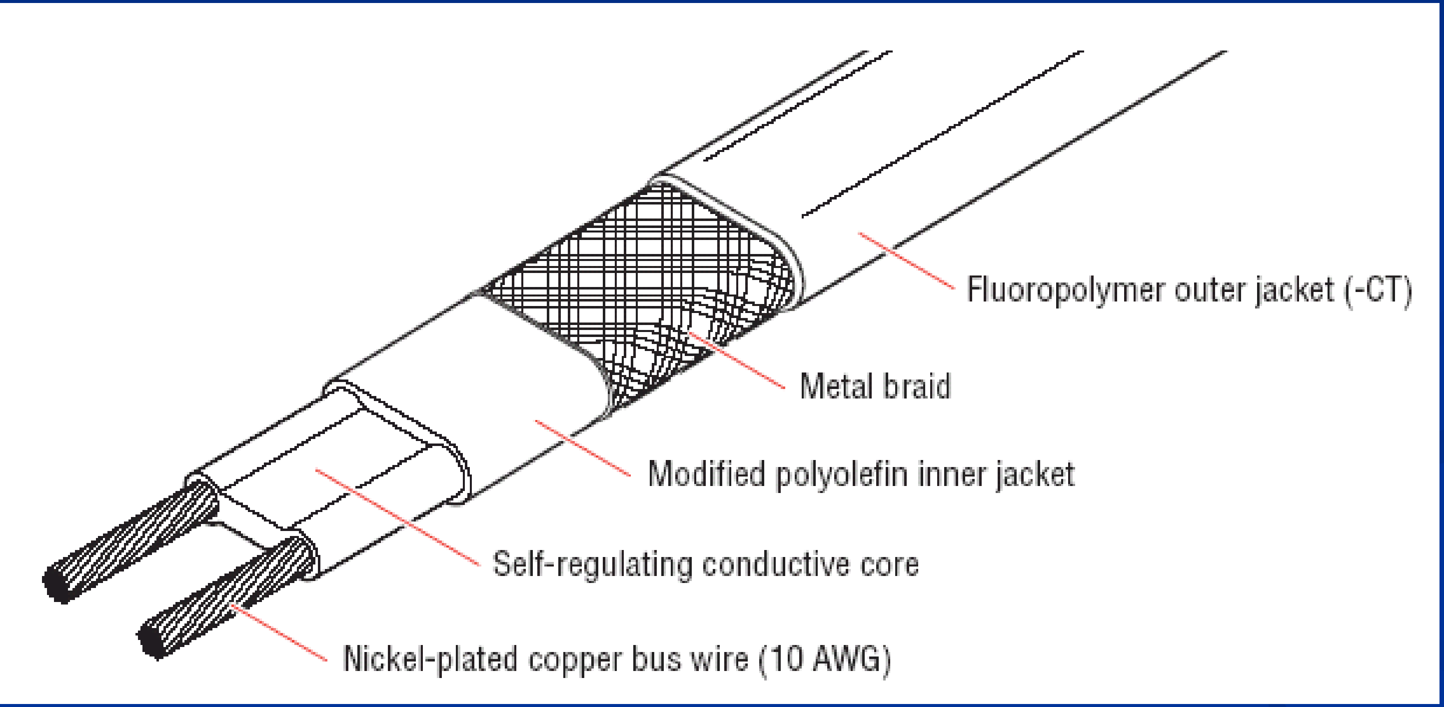
Product Dosimetry
PTC Materials

Positive Temperature Coefficient Materials

+ Self-limiting polymeric heaters
  - polyethylene dosimeters
  - resistance versus temperature test (R-T test)
  - gel content (rarely used)

+ Polyswitch® resettable fuses
  - polyethylene dosimeters
  - resistance cycle testing

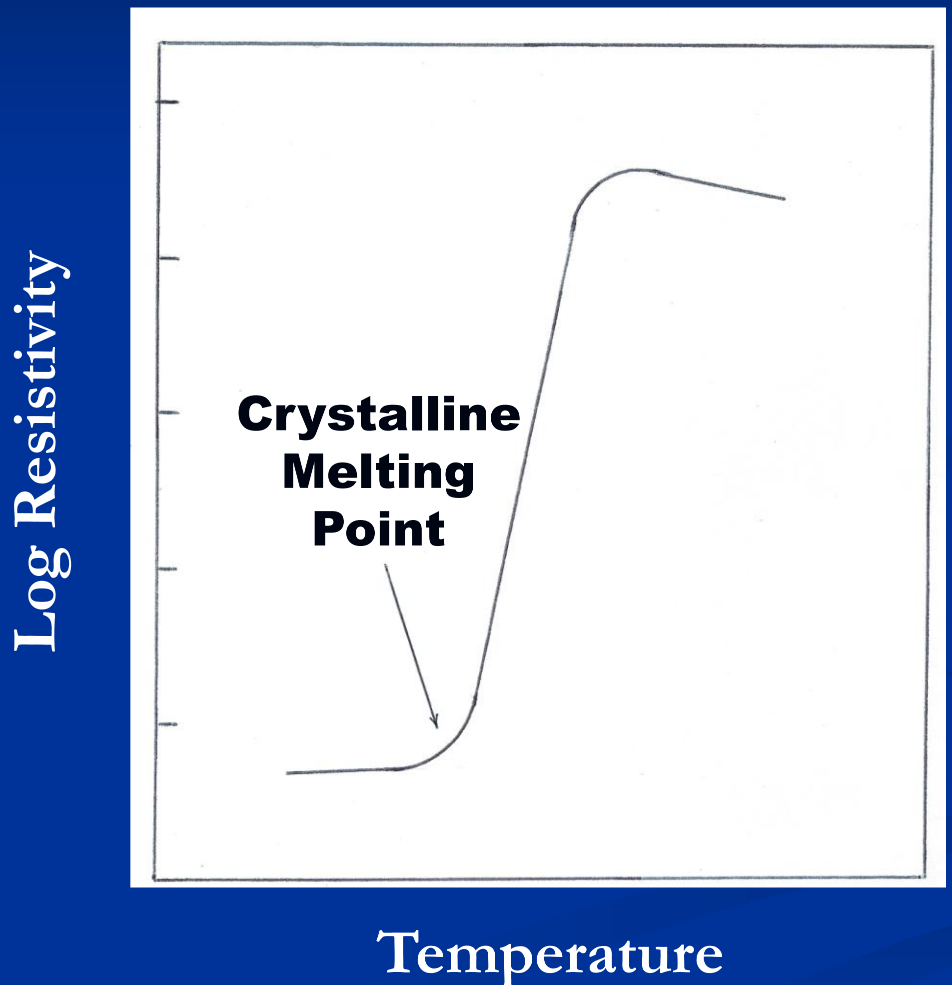
Product Dosimetry Chemelex® PTC Heaters



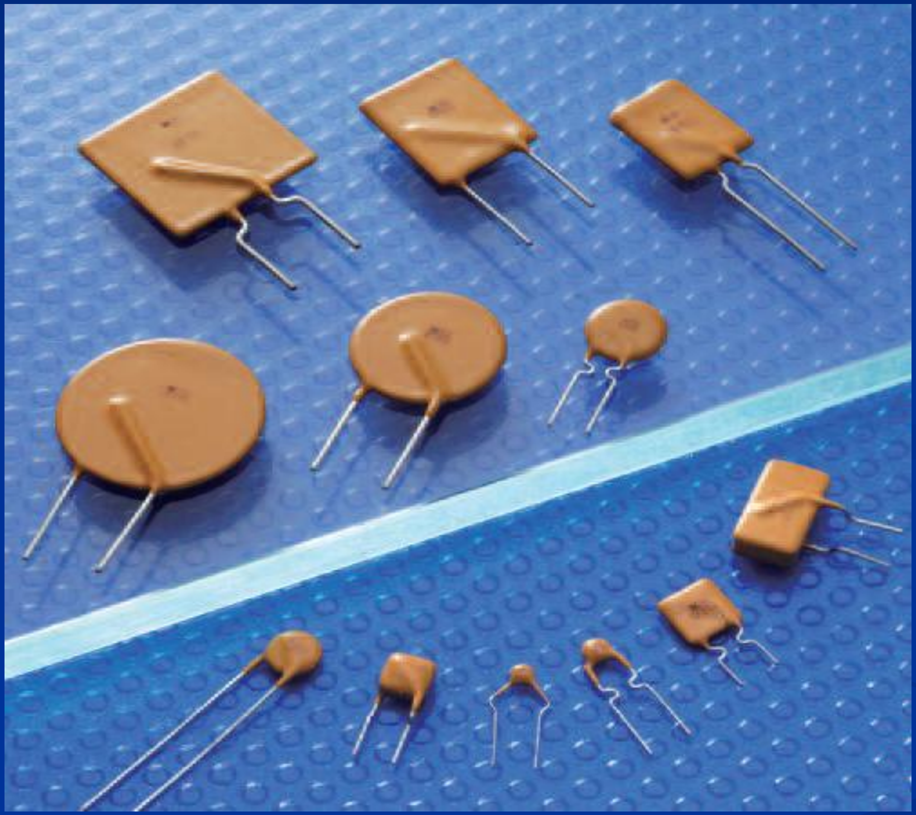
Use PE dosimeters

Resistance versus temperature testing

Product Dosimetry
Chemelex® PTC Heaters



Product Dosimetry
Polyswitch® Resettable Fuses



Use PE dosimeters

Product resistance cycling testing

Conclusions

+ Polyethylene is used as the base raw materials in at least half of all of the industrial electron beam processing applications through-out the world.

+ Polyethylene sheet is used as a reference dosimeter by a multi-site industrial electron beam user to control its EB processing.

+ Product performance can be correlated to PE irradiation responses and help standardize multi-site manufacturing operations.