

NEW MULTI PURPOSE LABORATORY
ELECTRON BEAM EQUIPMENT

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B. Laurell, E. Föll
Electron Crosslinking AB
Skyttevägen 42
SE-302 44 Halmstad
Sweden

1. INTRODUCTION

More environmental protection requirements force users of solvent-based coatings to look for an environmentally safe procedure that emits considerably less solvents or cracked products into the atmosphere and water.

Also the new EC Super directive regarding migration of less than 10 ppb into the food will force the packaging producer to look for new technologies. [28]

The chemistry has new groups of products to offer:

- Systems with low content of solvents and with high solid contents
- Coating materials containing water
- Coatings with solid resin in powder form
- 100 % solid systems.

A common feature of the first three methods is that heat is required for the formation of dry coating film.

Interesting techniques which do not induce these disadvantages of thermal drying are the radiation curing techniques methods – in particular those involving ionising radiation such as ultraviolet or electron-beam curing.

Radiation Curing

- | | |
|------------------------|----------------------------|
| - IR Infra-red | System containing solvents |
| - Micro-wave | Water-based system |
| - UV Ultraviolet light | 100 % system |
| - EB Electron Beam | 100 % system |

UV-ultraviolet light can be used without problems for curing in those places that are accessible to "UV-light", i.e. the layers, which the radiation must pass through, must be thin and transparent at the appropriate wavelengths. The formulation will contain photo initiators and other absorbing materials like pigment and matting agents.

During the curing process there should be no emission of harmful substances into the atmosphere, water or into the food. In addition, after the curing process is over, there should be no odour emissions from the surface.

These requirements have already given Electron Beam curing (EB) the focus of even more attention, not least as this technology has been used for a wide variety of applications in recent years due to the many other benefits it offers.

EB is the abbreviation for an environmentally safe, heat and solvent free technique: **Electron-Beam** curing.

Nevertheless, success is only possible if the user, the chemical supplier and the plant manufacturer collaborate fully - not only during the planning phase but also during the construction of the curing line and later during initial operation.

2. THE ACCELERATOR

The functioning of the electron beam accelerator can best be compared with the cathode ray tube of a TV. Fig. 1.

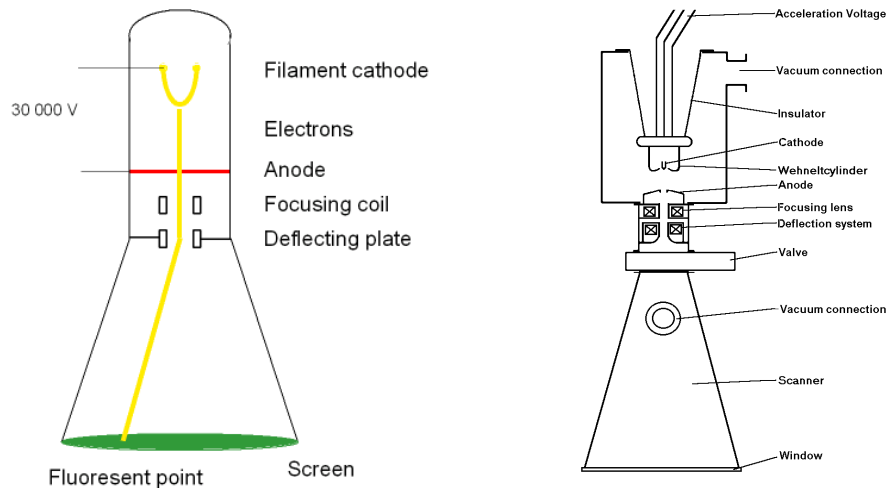


Fig. 1. Cathode ray tube and the Electron Crosslinking electron beam accelerator

The production of free electrons in vacuum is properly familiar to us all from the cathode ray tube and the television tube. A tungsten cathode heated in high vacuum by an electrical current makes free electrons available on its surface and these are accelerated to the anode. By suitable design of the cathode and anode (with a linear accelerator the anode is the electron-beam exit window) it is possible to focus the electron so that it is accelerated through an opening in the anode in the field-free space of the vacuum. In a TV set the electrons (negatively charged particles) are accelerated by a high negative voltage towards the anode and then deflected to the screen, or to the electron-beam exit window in the electron-beam accelerator. In the accelerator these electrons then emerge from the vacuum through a thin piece of titanium foil into the air or an inert gas where they can act upon the material.

A classic triode system is used for generating and forming a beam. A tungsten heating cathode, Wehnelt cylinder and an anode -with focussing lens and electron-beam deflection system- together form one unit (fig. 1). Absolutely linear current signals controlling the beam deflection in two perpendicular directions. Scanning frequencies are over 800 Hz.

The electron-beam exit window is designed to have a large surface area. A 7 to 20 microns thick titanium foil is supported on the vacuum side by means of a special construction. Cooling is effected by means of water and convection through this supportive copper plate. No additional window cooling from the outside is necessary by blowing either air or inertgas. This considerably simplifies window cooling and the inerting of the process.

The large area window has a standard length in conveyer direction of 100 mm or 220 mm. The working width is adjusted correspondingly to the object to be irradiated.

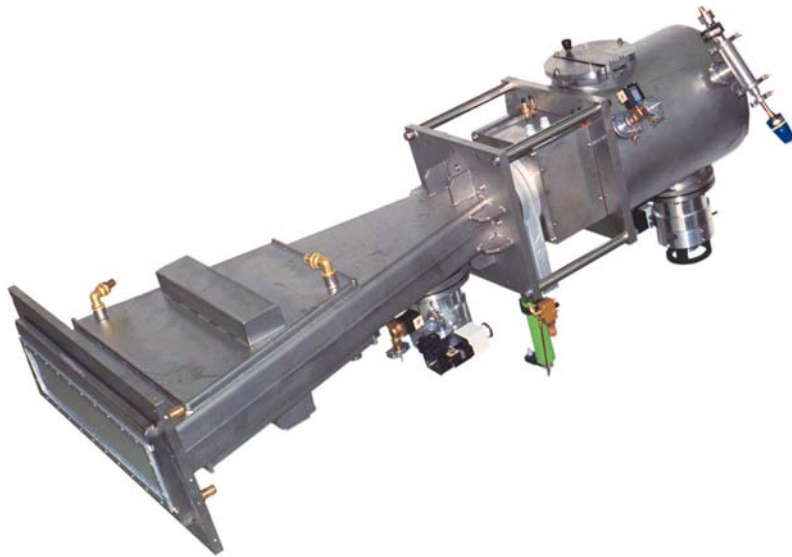


Fig. 2. Picture of Electron Beam Accelerator, 250 kV accelerating voltage and 1,20 m working widths.

Operating characteristics

- | | |
|--|-----------------|
| - Acceleration voltage | 80 - 300 kV |
| - Electron current | 0- 200 mA |
| - Working width | - 2000 mm |
| - Speed of web at 10 kGy | up to 900 m/min |
| - Distribution of dosage over working width | $< \pm 5 \%$ |
| - No gas cooling of the electron exit window necessary. | |
| - The accelerator can be installed in any position whatsoever. | |
| - No measurable radiation outside the x-ray shielding. | |

The accelerator is running at 24-hours operation in many different types of applications. It is especially distinguished by low set-up times following initial installation and service. Its uncomplicated construction combined with a control system for automatic process control enables the operator to quick and easy replacement of cathode and exit window without assistance from the supplier. Replacing wearing parts requires in total less than one hour.

3. EFFECTS OF ELECTRON BEAMS

Electrons are generated in vacuum and accelerated, then proceed through a titanium foil from vacuum to normal atmosphere and penetrate into the material with a range up to r_0 .

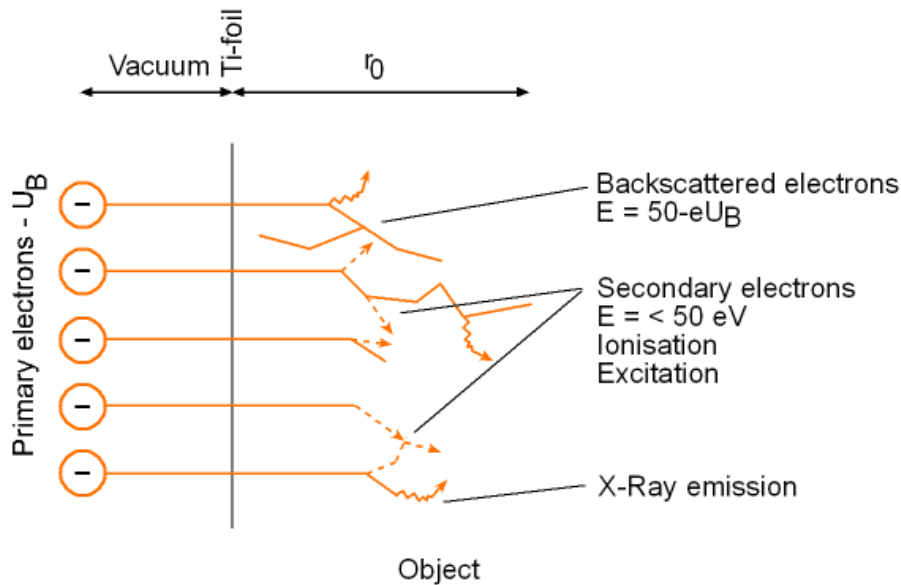


Fig 3. Electron interaction with matter (schematically)

After a distance of r_0 all electrons are retracted due to interaction with the material.

- Here Primary electrons / Backscattered electrons are generated. Their energy exceeds 50 eV and has a maximum corresponding to the accelerating voltage. The fast primary and backscattered electrons do not lead to chemical reactions. Their activation cross section is too low – they cannot be caught by molecules and thus do not lead to radical formation, ionisation or excitation.

- Important to us are the Secondary electrons at energy levels between 3 and 50 eV. They are slow enough, i.e. their activation cross section is large enough to ionise molecules and to form radicals. Very slow electrons less than 3 eV only induce Excitation. Ultimately we only need the fast electrons to generate secondary electrons at the location outside the vacuum and/or deep in material.

- A negative by-product of retarding accelerated electrons is the X-ray emission. Its energy cannot exceed the primary electron energy. This means that the electron-beam accelerator and the irradiation zone have to be shielded to prevent X-ray emission.

4. ENERGY TRANSFER

The transfer of energy from the electron beam into material is specified completely by four parameters:

- Depth of penetration
- Absorbed dose
- Beam uniformity
- Throughput

4.1 Depth of penetration

Penetrating power of the electron beam is related to the accelerating voltage and the density of the processed material. Higher voltage causes deeper penetration, and denser material reduces the depth of penetration. The Depth Dose Curves (Fig 4) are convenient aids for estimating the penetration depth. These curves show the penetration for different accelerating voltage to the depth of penetration in a material with mass density equal to that of water, i.e. $\rho = 1 \text{ g/cm}^3$.

Penetration into materials of different density can be estimated by multiplying the penetration depth, found from the normalized curves, by the ratio of the density of water to the density of the material. For example, a 200 kV beam will have a 50 % dose point at 0,246 mm in water and 0,123 mm in a material twice as dense ($\rho = 2 \text{ g/cm}^3$).

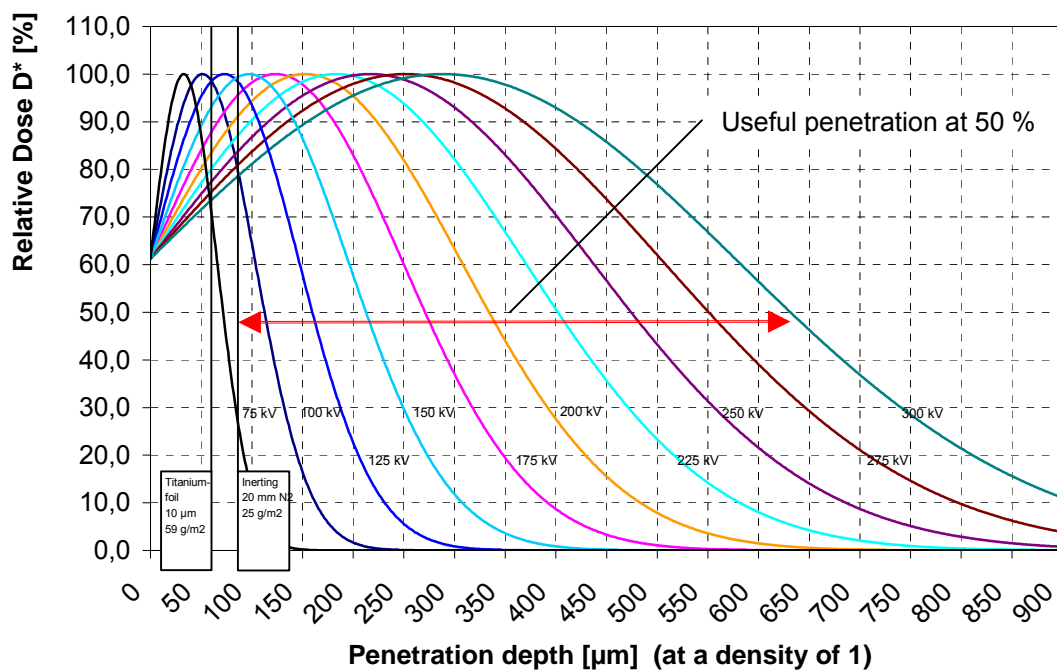


Fig 4. Range of penetration with different accelerating voltage 13 µm titanium foil and 20 mm of Nitrogen. [22]

At accelerating voltages of 150, 180 and 250 kV respective curing depth of 86, 138 and 277 g/m^2 are achieved at 80 % ionisation.

Experienced values for industrial accelerating voltages are as follows:

- 80 – 150 keV thin layers in the field of printing inks or silicon-release materials, sterilisation
- 165 – 180 keV furniture foil, pressure-sensitive adhesives
- 180 – 250 keV boards, parquet, panels, lamination
- 250 – 300 keV composite

4.2 Absorbed Dose

Absorbed dose is defined as the amount of energy deposited into a specified mass of material. The unit of absorbed dose is gray (Gy), defined as the number of joules (J) of energy deposited into 1 kilogram (kg) of material. An older, but frequently used unit, is megarad (Mrad).

$$1 \text{ Gy} = 1 \text{ J/kg}$$

$$1 \text{ kGy} = 1 \text{ kJ/kg}$$

$$1 \text{ Mrad} = 10 \text{ kGy} = 10 \text{ J/g} = 2,4 \text{ cal/g}$$

- | | |
|--------------------------------|-----------|
| - Heating of water 1 degree | 1 cal/g |
| - Evaporation of water | 540 cal/g |
| - EB-curing of lacquer approx. | 10 cal/g |

At a fixed electron accelerating voltage, the dose is directly proportional to the electron beam current. The dose D [kGy] is proportional to electron current I [mA] and inverse to web speed v [m/min] as follows:

$$D = k \times \frac{I}{v}$$

the k factor is depends on the equipment and the accelerating voltage.

The formula above shows:

- dose and electron current are directly proportional
- if the ratio of electron current and speed are kept constant the dose is constant including start up and shut down of the plant
- the accelerator uses only the quantity of power from the main supply needed for the used web speed
- quality improvements

Typical values of the dose needed for practical applications are:

- Drying/curing of inks and coatings 15-30 kGy
- Crosslinking of plastic films 25-150 kGy
- Sterilization of medical products 7.5-35 kGy
- The certified dose to sterilize: 7 log decrease is 25 kGy [27]

4.3 Beam Uniformity

Beam uniformity is a direct function of how the electron beam is distributed over the working width. It is specified as a percentage deviation from the average value, e.g. 20 kGy \pm 10%. In general, Electron Crosslinking Accelerator provides a uniformity better than \pm 5%; many applications can tolerate variations of \pm 10% or more.

4.4 Throughput

Throughput is a measure of the energy deposition rate and relates directly to the amount of material that can be processed within a given time interval. It is measured in kilogray per second, abbreviated kGy/s.

An Accelerator specified to 10 000 kGy m/min can provide a dose of 25 kGy when the web speed is 400 m/min, or 50 kGy at 200 m/min, etc. The processor will automatically adjust the beam intensity as the web speed changes so that the dose remains constant.

5. APPLICATIONS

In all applications the Electron Beam Accelerator itself remains the same but the handling system differs:

- material in solid form, as sheet, board, panels etc.
- flexible materials, roll to roll
- laboratory equipment

5.1 Solid materials

In the surface converting of solid materials the EB-technology is successfully used in the following operating fields:

Curing of top lacquer on doors [1], [2]

All-around curing of coated profiles [3], [4]

Curing of the coating on raw boards in the wood industry [5], [6]

Curing of the coating on architectural claddings for outside applications [7], [8]

Curing of the coating on wood-cement boards for outside and inside application [9]

Curing of impregnation and top lacquer on laminated boards

Curing of coated edges and panels in the wood and laminate industry [10]

Curing of coatings on MDF boards (Medium-Density-Fibre-board) [11], [12], [13]

Curing of coatings on three-dimensional parts e.g. rims and pumps housings.

Sterilization

5.2 Flexible materials

In the surface converting of flexible materials the EB-technology is successfully used in the following operating fields:

- Vulcanisation or crosslinking of pressure-sensitive adhesives [14]
- Curing of high-gloss coating of special paper (e.g. photographic paper) [15], [16], [17]
- Curing of release coatings
- Curing of web offset printing inks, finishing varnishes [18], [21]
- Crosslinking of films and foils
- Production of antistatic finish
- Crosslinking of flock adhesives
- Curing of intaglio prints [18], [19], [20]
- Post-crosslinking of binding agents of magnetic materials
- Metallizing of paper, e.g. curing of basic lacquer and adhesives for selective or plane transfer metallizing as well as curing of overprints
- Curing of metal coating from roll to roll (coil coating)
- Stabilisation of rubber raw materials by partial vulcanisation
- Crosslinking of laminating adhesives
- Crosslinking of thin insulation of wire and cables
- Colouring of textiles
- Sterilization

5.3 Laboratory equipment

Electron Crosslinking has developed a new compact multi purpose laboratory equipment that can be used in many different types of laboratories used by the chemical industry as well as the end user for the development of new processes and for quality insurance in production. Fig. 5 shows the EC-LAB 400 basic laboratory equipment with batch transport suitable for laboratory work.

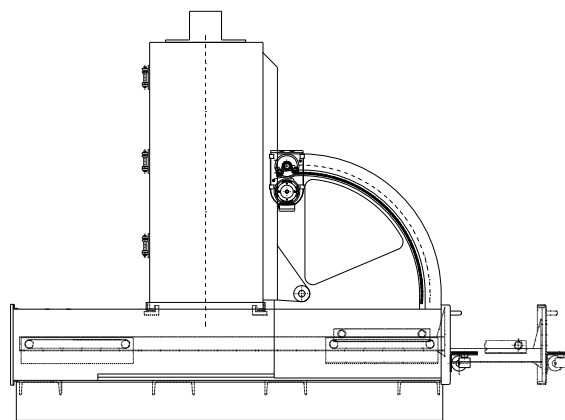


Fig. 5. EC-LAB 400 basic unit for batch transport (schematically).

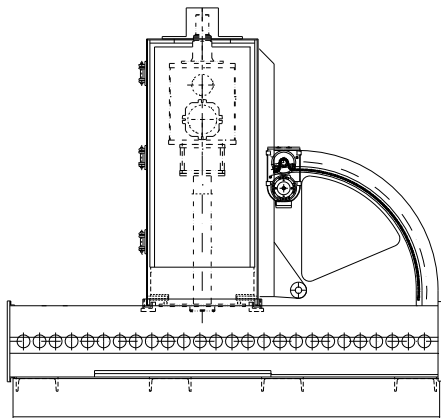


Fig. 6. EC-LAB 400 unit for roller transport (schematically).

The equipment has a modular design and can easily be modified for many different applications; here is one option for continuous roller transport for adaptation in other processes.

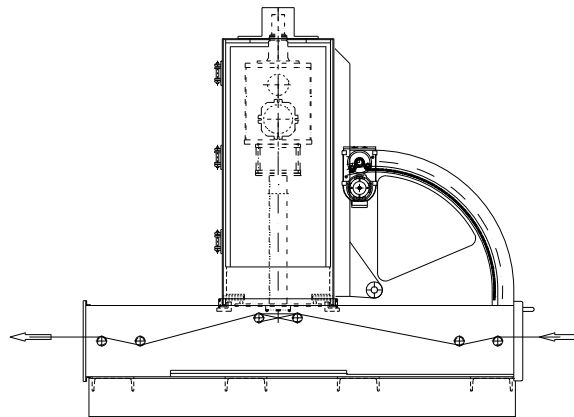


Fig. 7. EC-LAB 400 unit for cable or fibre transport (schematically).

An interesting system is the option for continuous irradiation of cables, fibres and composites that will open many new possibilities.

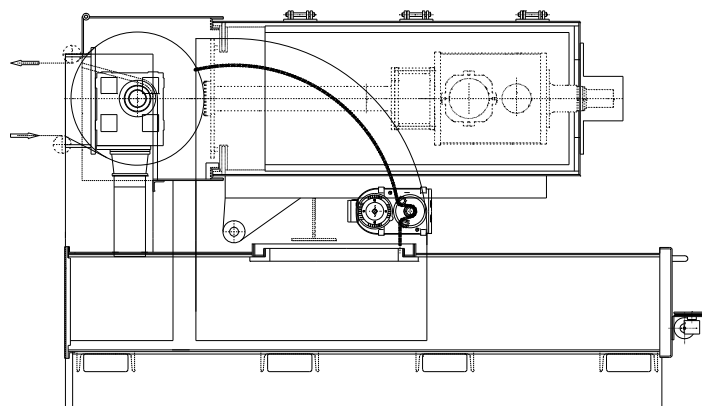


Fig. 8. EC-LAB 400 unit for web transport with cooling drum (schematically).

The electron accelerator is positioned horizontally and is aimed towards a drum. The drum can be cooled or heated.

Together with a large-surface electron-beam exit window, the drum can be heated up to temperatures of 100°C. The exit window can be operated at temperatures of up to 70°C.

This is an interesting variation of the application technique, particularly with respect to monomer-free materials applied at higher temperatures, and according to the chemicals used, crosslinked in a hot state.

The drum serves to guide the material during the hardening or vulcanisation process and is indispensable for hardening of the lacquer (especially on thin, highly flexible substrates), fig. 8.

Use of a drum permits inertisation of the process zone.

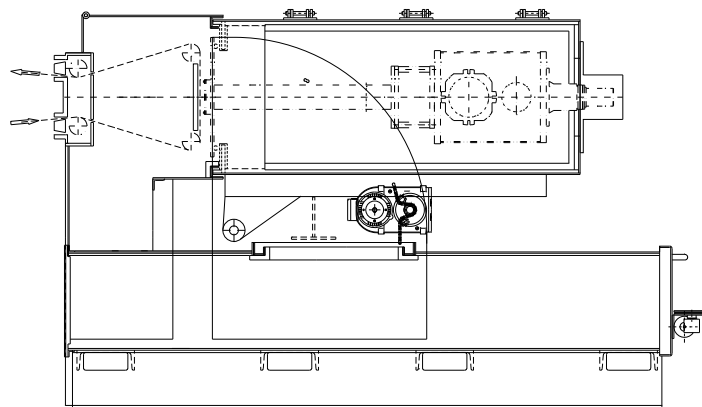


Fig. 9. EC-LAB 400 unit for web transport with cooling plate (schematically).

Typical data for EC-LAB 400

Accelerating voltage:	80 – 300 keV
Electron current:	0 – 30 mA
Working width:	max 400 mm
Throughput:	4500 kGy m/min
Exit window:	Titanium
Thickness:	7 – 20 μm
Cassette speed:	5 – 30 m/min
Max size (LxWxH):	600 x 400 x 250 mm
Web speed:	0 – 150 m/min
Max web width:	450 mm

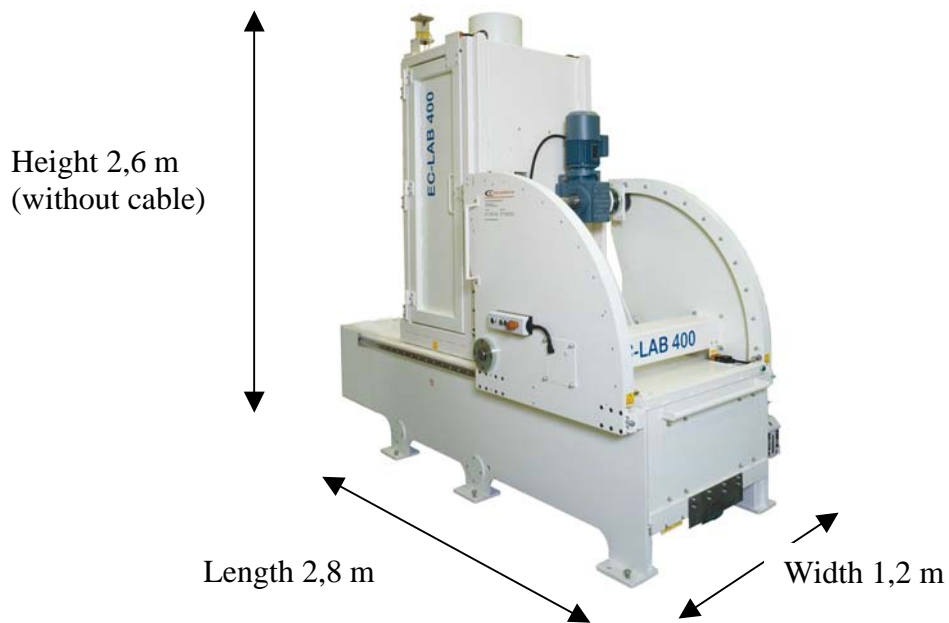


Fig. 10. EC-LAB 400 batch system.

This picture shows the basic design with integrated batch system for the treatment of material in the range of 80 – 300 kV. The transport speed is adjustable from 5 to 30 m/min. With a working width of 400 mm most laboratory needs are covered.



Fig. 11. EC-LAB 400 accelerator with pump system (80 – 300 kV, 5-30 m/min, 400 mm).

The construction of the accelerator is based on the system that is used for 24 hours operation. Maintenance is easy and usually carried out by the customer.

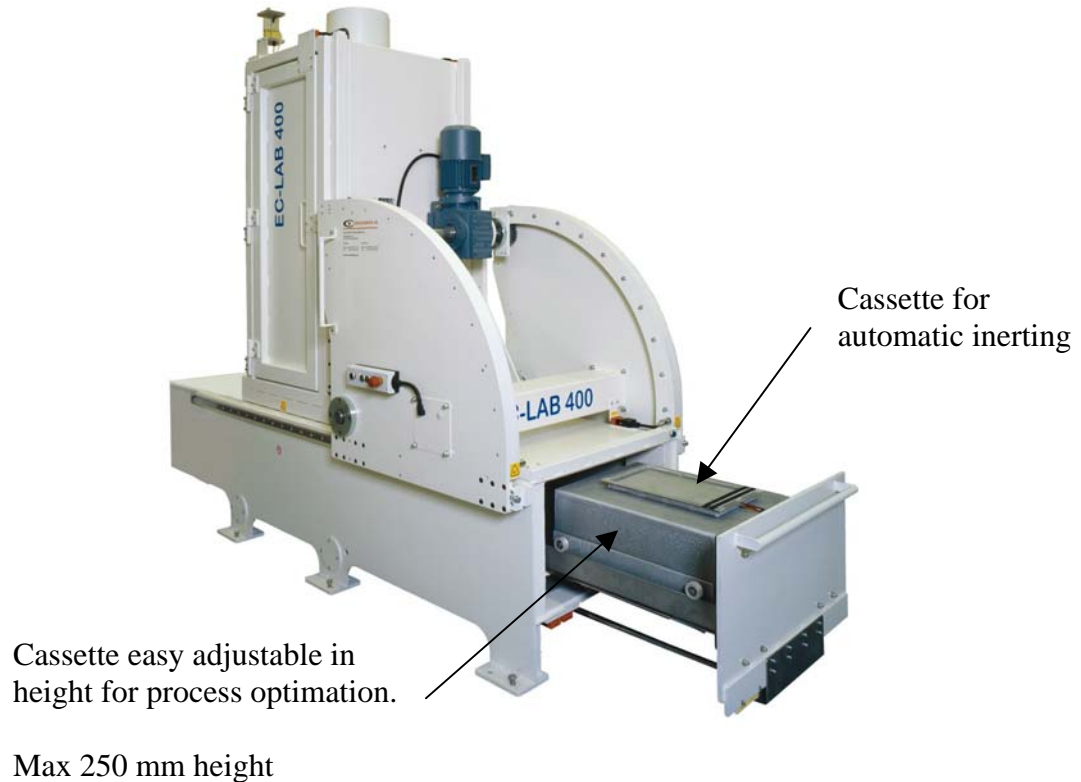


Fig. 12. EC-LAB 400 in load position for the cassette (80–300 kV, 5-30 m/min, 400 mm).

The cassette can be flushed with Nitrogen or other gases on customer request. After closing the door, the accelerator will start the high voltage and the beam current, and after the inertisation the transport system will start to move the cassette under the accelerator. The set point for the accelerating voltage, beam current or dose and transport speed is easy to adjust on the screen. After the treatment the batch transport system automatically returns to the start position and the door can be opened. The system is ready for the next treatment.

The cassette is available for both 3-D and 2-D products. Maximum working height for the cassette is 250 mm. The transport system is easily adjustable in height for process optimisation.



Fig. 13. EC-LAB 400 with both batch and roller option (80-300 kV, 0-150 m/min, 400 mm).



Fig. 14. EC-LAB 400 with both batch and roller option closed position.



Fig. 15. EC-LAB 400 unit with combined batch and roll-to-roll features in service position.

The picture shows the drum option and accelerator in service position for easy feeding in the web. The design is made for quick and easy cleaning of the drum. The drum can be exchanged with drums with different surfaces and structures for many applications.



Fig. 16. EC-LAB 400 system with combined batch and roll-to-roll features service side.

6. EB -CURING

Advantages of Electron Beam curing:

- Environmentally friendly due to a 100 %-solid system. EB generates absolutely no emissions.
- No substrate heating.
- Low energy consumption.
- Substantial production increase compared to conventional heat-treatment methods and UV-technology, also with pigmented layers.
- Immediate further treatment of converted products without post curing.
- Low space requirement. Integration into existing production processes without any problems.
- Exact repeatability of production conditions is obtained due to high dose accuracy. There is also no wastage when starting up and shutting down the plant.

7. SUMMARY

Electron-Beam curing has overcome its limits and is heading to new applications.

Electron-Beam has a growing attention as an eco-efficient technology.

The growth rate in Europe for the Radiation Curing market is estimated to 9 % yearly.

In order to carry this technology on to success, good cooperation between customer, chemistry and plant manufacturers is necessary.

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