POLYMERIC MATERIALS RESEARCH WITH CYCLOTRONS

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Ion beams from cyclotrons as a source of radiation with extremely high linear energy transfer (LET) are considered. Basic processes of energy dissipation in heavy ion tracks are reviewed and the main parameters characterizing the effects of accelerated heavy ions on matter are given in relation to the effects of low-LET radiation. Modification of polymers by using cyclotron beams is discussed with the emphasis on some most recent results. Track membrane production is presented in more detail as an example of industrial application of cyclotron beams.

1 Introduction

Ion beam processing has become common tool for the analysis, synthesis and modification of materials. It is now used in a number of applications related to various materials: metals, semiconductors, superconductors, insulators. Among other insulators, polymers occupy a prominent place due to some special properties and a wide use of high molecular weight substances in practice.

The processing of polymers by using high energy particle beams from cyclotrons is not yet a routine procedure like, for instance, ion implantation in the keV energy range - an area giving a huge amount of publications [1,2]. At present, the number of applications of accelerated ions with the specific energy of several MeV/u for the polymeric materials research and modification is sufficiently smaller. Nevertheless, the study of the effects induced by high energy ions on polymers attracts growing attention both for the fundamental interest in non-conventional irradiation effects and for potential technological applications which is illustrated by the proceedings of some recent conferences [3-6]. Moreover, there exist applications on really industrial scale in this domain and we shall pay proper attention to them in the present report.

Cyclotrons provide particle beams in a wide range of atomic numbers - from protons to uranium ions. This report aims at focusing on the effects of heavy ions, i.e. particles heavier than He, although sometimes the data related to protons and alpha particles will be included for comparison.

2 What is special about polymers?

Polymers are solid high-molecular substances with the molecular masses from thousands to millions. The long chain structure of polymeric molecules determines a strong difference between radiolysis of polymers and small molecules. Let us explain this by a simple example of a typical polymer with the molecular mass of 3×10^5. If the polymer has moderate sensitivity to the ionizing radiation, the radiochemical yield of the radiation-induced changes is about 1 per 100 eV of absorbed energy. By introducing a relatively low dose of 10 kGy (10^4 Joules per kg) into the polymer one creates ca. 6×10^{17} radiolytic events in a 1 g sample which means that about 30% of the macromolecules underwent radiolytic changes. Such changes can have a significant effect on the macroscopic properties of the polymer whereas the influence of a 10 kGy dose on metals, ceramics, glasses and other low-molecular weight bodies is negligible.

Another important feature of polymers is that, in contrast to most inorganic solids, the physical phase of radiolytic process is followed by a "mild" chemical stage. The "mild" chemical reactions can last from seconds to years and lead to further changes in the irradiated material both on microscopic and macroscopic scales. The environment effects are very important during the phase of chemical transformation. The final state of the polymer depends on the ambient temperature, atmosphere (first of all on the presence of oxygen), pressure, etc.

3 Cyclotron beams from the point of view of the radiation chemistry

Dramatic changes in the radiation induced damage processes may occur if heavy particle beams from cyclotrons are used for the irradiation, instead of conventional-condition irradiations such as electrons or gamma rays. The reason is a very high value of electronic stopping power of the ions, dE/dx, which induces generation of a great number of electron-hole pairs in a local vicinity along the ion path. From the point of view of the radiation chemistry, the most important parameter characterizing the quality of radiation is the linear energy transfer (LET) which in the case of the

Table: Parameters of typical radiation sources used for the treatment of polymers

	LET, MeV/μm	dD/dt, Gy/s	Penetration depth in polymers
1.25 MeV gamma rays from ^{60}Co source	2×10^{-4}	1 - 100 [7]	centimeters
1 MeV electrons from 10 kW accelerator, focused beam	2×10^{-4}	2.5×10^{6} [7]	4 - 5 mm
1 MeV/u Xe ions from the U-300 cyclotron (FLNR JINR)	10	10^{7} (focused beam) [8,9] 5×10^{4} (scanning beam) [8,9]	15-20 μm

accelerated heavy ions coincides with dE/dx. Another important parameter is the dose rate dD/dt, i.e. the absorbed dose in a unit time. The balance between chemical reactions of primary and secondary species in polymers depends to a great extent on the dE/dx and dD/dt values.

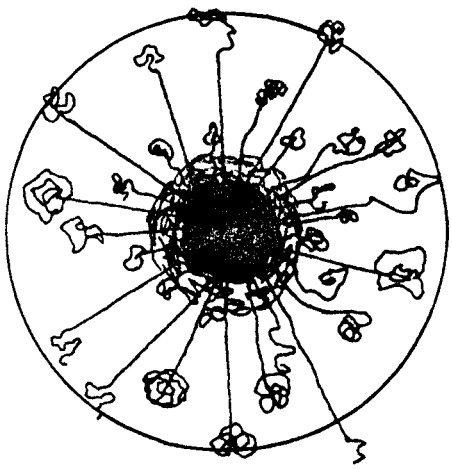

Fig. 1. Schematic model of a heavy ion track (cross-sectional view). The core and penumbra radii are shown [12].

The table shows typical data for various kinds of radiation sources to illustrate properties and potentialities of cyclotron beams. It is obvious that, due to the short range of heavy ions in matter, cyclotron beams can be used for the surface treatment and, only in the case of filmy materials, for the bulk modification. Present-day cyclotrons provide beams with the ion energies in the order of 10 MeV/u and even 100 MeV/u which expands the treatments depth up to millimeters but usually it is accompanied by lower intensities and dose rates than shown in the table.

Speaking of the accelerated ion beams as tools for materials modification, we have to mention that there are distinct differences between the requirements for the nuclear physics experiment conditions and those for the materials research. In general, energy spread and emittance of the ion beam are not so significant for the radiation induced damage studies. It is very important to control the dose, dose rate, homogeneity, irradiation temperature, atmosphere, etc. Sometimes the ion current should be extremely constant in time (for instance, in precise track membrane production).

4 Track formation in polymers: physics and chemistry

In fact, all kinds of modifications of polymers by heavy ion bombardment are based on the creation of the "tracks" in the material, i.e. on the "tracking" process. Initially the tracks in polymers and other insulators were used to detect heavy particles [10]. Slowly the concept of a passive diagnostic technique was replaced by that of an active structural tool for imprinting structure onto solids [11]. Therefore, the track formation mechanism and the nature of the tracks are the subjects of a key importance for our survey.

Several mechanisms are responsible for degrading the kinetic energy of a heavy ion in matter but for the specific energy range between 1 and 10 MeV/u the energy loss through electronic interaction (excitation and ionization) is the only important mechanism. After Chatterjee and Magee, there are two gross categories of interactions: glancing and knock-on [12]. Consequently, the tracks of the high energy particles are composed of two parts designated as "core" and "penumbra". The core is created by the glancing collisions with excitations less than 100 eV. The penumbra (or "halo") is composed of the knock-on electrons. Fig. 1 presents a cross-sectional view of a track 10^{-15} s after the passage of the heavy ion. One can see the core and the penumbra as the zones with very different densities of the electron tracks. In the core the electron tracks overlap whereas the penumbra consists of the individual electron tracks.

As a result, a highly positively charged plasma cloud around the ion path is created (see Fig. 2). The cloud "explodes" via the electrostatic repulsion of the positive ions in the track core and initiates an atomic collision cascade. This process is usually referred to as the "Coulomb explosion" [10]. Chemically active species generated in the electronic collisions and atomic collisions diffuse into the surrounding space and undergo secondary

reactions determining the changes of the polymer structure in the track halo.

After completing all the phases of energy dissipation - rapid and slow - we have a damaged zone with different density, different molecular mass and containing new chemical bonds and groups. This zone is capable of preferential etching.

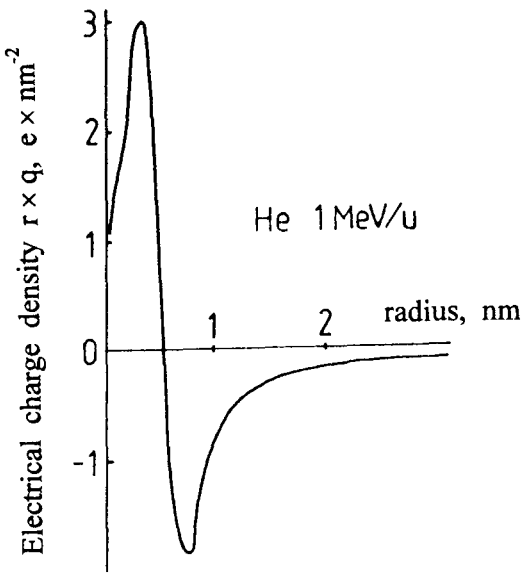

Fig. 2: Transient (10^{-14} -10^{-13} s) electrical charge distribution in a heavy particle track in insulator (after A.G.Weisburd and D.I.Weisburd [13])

Under certain conditions some features of the track structure in a polymer can be studied by performing conductometric measurements of the effective pore size as a function of time during the chemical etching. A typical result for a single heavy ion track etching in a thin film of polyethyleneterephthalate is shown in Fig. 3. The track core manifests itself as a sharp rise of the pore diameter in the range of several nanometers. After the dissolution of the track core, the radial etch rate decreases to a minimum and then rises again slowly approaching the etch rate of the virgin polymer. The region of the low etch rate is interpreted as a zone of preferential cross linking in the track halo extending to radii of tens nanometers [14]. It has been found that the size of the preferentially etchable track core depends only slightly on the chemical structure of the polymer which means that the core-size determining mechanism is rather a physical one [14,15]. At the same time the chemical activity of the material in the track is strongly influenced by the secondary chemical reactions.

5 Studies of high-LET effects with cyclotron beams

In polymers subjected to ionizing radiation the following chemical modifications are observed: main-chain cleavage and/or cross-linking, formation of unsaturation (double and triple bonds) and gas evolution. The early studies of heavy ion effects on polymers were based on the concept that the balance between different modifications depends on the LET but there is no essential difference between the primary radiolysis products generated by gamma rays/electrons and heavy particles. Thus, mainly *quantitative* differences were the subject of a number of research works performed with polymers irradiated on cyclotrons and other accelerators.

To my knowledge, the first attempt to perform a systematic study of high-LET effects with cyclotrons was made in the late sixties. The cyclotron of the Institute of Nuclear Research (Kiev) was used a source of 28 MeV alpha particles and 14 MeV deuterons which enabled the authors of [16] to study the LET effects in the range up to 0.3 MeV/μm. The authors observed a marked increase of the radiochemical yields of cross links in polystyrene (PSt), a strong yield decrease of the main-chain ruptures in polymethylmethacrilate (PMMA) and no changes of the degradation yield in cellulose diacetate with the growing dE/dx. The above mentioned work was followed by the series of papers [17-19] devoted to a number of various polymers. It was concluded that the LET effects are caused by the spatial distribution of the radiolysis intermediates in the tracks. Polymers were categorized according to their sensitivity to the increasing dE/dx. The polymers (first of all aliphatic ones) showing the same radiochemical yield for the low-LET radiation and heavy particles were classified as systems with a very fast energy migration leading to a homogeneous distribution of active species in the volume. The polymers with the pronounced LET effects were placed into another group.

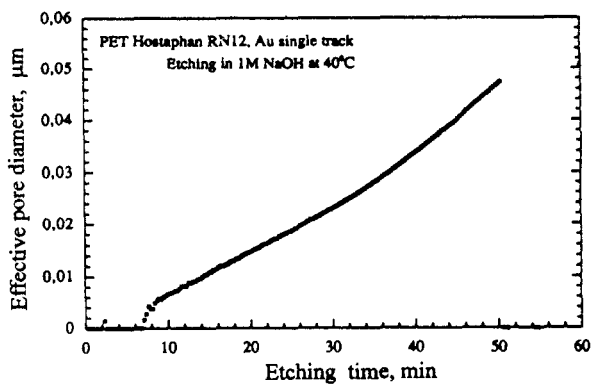

Fig. 3: Effective pore diameter as a function of etching time for the sample with a single heavy ion track

Sufficiently heavier energetic particles were used in the experiments performed with the use of ion beams from the Alice cyclotron at Orsay. A.Chambaudet with co-authors carried out IR and ESR studies of some typical polymeric detectors irradiated by electrons and Ar and Kr ions with the energies of several MeV/u [20,21]. The infrared spectra did not show any difference between the two types of irradiations implying therefore that there is no

difference in the nature of the permanent chemical transformations induced by the heavy ions or electron bombardment. On the other hand, the ESR measurements revealed the production of carbon-like radicals in the ion tracks which were not observed in the electron-irradiated samples. It was concluded that the carbon-like radicals (similar to those produced by polymer pyrolysis) correlate with the latent track formation.

The infrared spectroscopy and visible spectroscopy combined with the scavenger technique were applied to the chemical track structure studies performed by the researchers from Limoge University [22-24]. Ion beams produced by the Alice (Orsay) and by the tandem at Saclay were used for the bombardment of cellulose derivatives. Estimates of the "chemical" track radii were obtained and compared with the theoretical predictions.

The accelerated Ne and Ar ion beams from VICKSI (HMI, Berlin) providing the LET as high as 0.4-2.3 MeV/μm were used in [25,26] to investigate the main-chain scission and cross-linking phenomena in several polymers. Some previous results of other authors were critically re-examined. It was confirmed that, for instance, PMMA appears to degrade less effectively with increasing LET. In contrast, PSt did not show any change of the cross-linking yield with changing dE/dx. The authors suggested that the change of the cross-linking efficiency of PSt observed earlier was rather a result of thermal effects and/or dose rate effects than that of high LET. If the dose rate and thermal effects are carefully avoided, dE/dx has no influence on the cross-linking of polystyrene, obviously, due to a very low yield of radicals acting like cross-linking precursors [26]. Apparently, this situation is not typical. Most polymers able to form cross links exhibit an increasing yield of the cross-linking under the bombardment with heavier particles [9,27]. Especially clear LET effects of this kind are observed in aromatic polymers [28].

Summarizing, we can conclude that the methods based on the measurements of bulk properties of heavy-ion irradiated polymers showed generally a *qualitative* similarity of the observed phenomena to those obtained with the low-LET irradiations. The similarity concerns the formation of the intermediates (electronically excited molecules, free ions and radicals). Obviously, the local intermediate concentration is very high in the tracks of heavy particles and, therefore, the observed quantitative LET effects should be considered to be due to the change of intermediate reaction probabilities which strongly depend on the local concentration.

6. Heavy ion effects on polymers: some newest results

Within the last few years the advancing to the region of very high dE/dx and the progress in experimental equipment gave some *qualitatively* new results worth of special note. The medium energy line facility at GANIL (Caen) was used to investigate the swift heavy ion effects in a wide range of projectile masses and dE/dx [29,30]. The changes in chemical structure were investigated by Fourier transform infrared spectroscopy (FTIR). The irradiations by MeV electrons were performed in very similar experimental conditions for comparison. The FTIR measurements indicated a significant increase of the yield of chain scission associated with unsaturated end-groups. Moreover, some specific modifications under the heavy ion bombardment were evidenced, i.e. modifications which are never observed with the use of electrons or γ-rays, namely the creation of alkyne and allene end groups:

$$\sim CH_2 - CH_2 \sim \rightarrow >C = C = CH_2$$
$$\sim CH_2 - CH_2 \sim \rightarrow \sim C \equiv CH$$

It is obvious that such modifications require "evaporation" of at least 3 neighboring hydrogen atoms simultaneously - an effect clearly appearing with increasing dE/dx.

Another interesting study was performed with the use of the cyclotron at Louvain-la-Neuve. Polycarbonate films and a model compound were irradiated by accelerated Ar and Kr ions and analyzed by gel permeation and high-performance liquid chromatography, electron spin resonance, thermogravimetry, ultraviolet and infrared spectroscopy [31]. A wide variety of low-molecular weight species produced from the model compound were identified. These results shed light on the very intricate modifications occurring in the tracks of highly ionizing particles and on the post-irradiation chemistry of the "tracked" material. Further experiments can be directed on the selective extraction of polymer radiolysis products from tracks [32,33] and subsequent analysis by means of complementary methods.

The next work to be mentioned was performed with non-polymeric target but relevant to polymers. Frozen methane was irradiated by proton and He ions from the cyclotron of Forschungszentrum Juelich [34,35]. After the successive warming to ambient temperature the residues were analyzed and identified. Long chain aliphatic and olefinic hydrocarbons, aromatic and polycyclic compounds were found among the radiolysis products. The experiments gave evidence for a fast multicenter combination of intermediate radicals formed by the secondary suprathermal carbon atoms from knock-on processes in one collision cascade. Thus, the synthesis of new and complex compounds in high energy heavy ion tracks compete with the pure radiolytic fragmentation of polymers [36,37]. These results underline the possible role of the non-equilibrium suprathermal chemistry in the prebiotic buildup of complex organic matter in space rich in energetic heavy particles.

Another prominent example of profound alterations occurring in the polymer structure is the buckminsterfullerene formation [38,39]. The C_{60} carbon molecules have been previously synthesized in flames, sparks, arcs, laser beams and under high dose-rate electron irradiation. A common feature is the high transient energy density followed by a rapid quench. Both these phases are

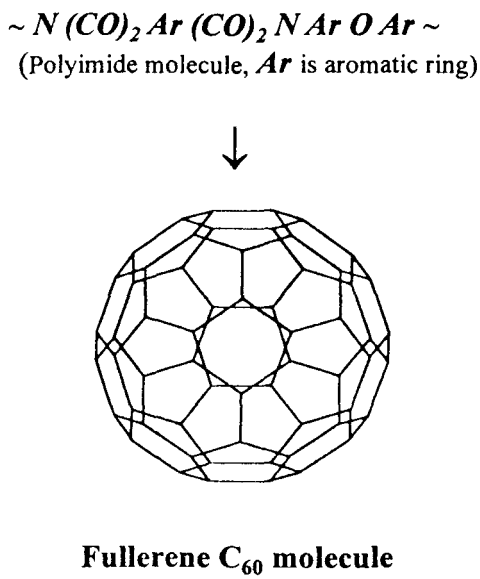

Fig. 4: Constructive transformation of polymer molecules in heavy ion tracks

present in the swift heavy ion track core: the extremely high energy pulse in a small volume and the quenching with an extremely high rate. Thus, the heavy ion track can be considered as a unique plasmochemical reactor providing a "reconstructive transformation" of matter [38]. Apparently, the probability of the transformation shown in Fig. 4 is very low but not zero [39]. It is now quite clear that ion beams can introduce order into solids and that molecular *construction* surpasses *destruction* processes above a certain energy threshold lying in the GeV total energy range.

7. Applications

7.1 Track membrane production

Manufacturing of microfiltration membranes by irradiation with accelerated particles has become an alternative to the method based on the bombardment by fission fragments in nuclear reactors [10]. One can outline the following advantages of the accelerated heavy ions:

1. There is no radioactive contamination of the polymeric foil and therefore there is no need to "cool" the irradiated material before chemical etching.
2. High intensity of ion beams from cyclotrons makes it possible to achieve higher pore densities in the membranes.
3. The identity of particles provides identity of tracks and their etching properties.

4. Due to the higher energy of accelerated ions, thicker films can be perforated including those exceeding the range of fission fragments.

5. The atomic number and the energy of the particles can be specified according to the requirements. One can also control the angle of incidence and use a parallel or non-parallel beam - depending on the required structure.

Efforts to use cyclotrons for the track membrane (TM) production were made at different laboratories (FLNR JINR in Dubna [8,40], GANIL in Caen [41], IPN in Orsay [42], CRC in Louvain-la-Neuve [43], RIKEN in Saitama [44]. The accelerators of other types than cyclotrons were also used for tracking the materials [11,45,46]. Let us consider the evolution of the heavy ion track membrane technology at the example of FLNR. The first track membranes were produced on heavy ion cyclotrons at FLNR JINR in Dubna in the early seventies. By 1980 the manufacturing procedure of polyethylene terephthalate (PET) membranes was elaborated and the first pilot machines for ion irradiation, ultraviolet treatment and chemical etching were constructed. During the next decade the facilities and technology were improved significantly. The xenon ion beams of high intensity from the U-300 cyclotron were used for the irradiation of polymeric films. Many attempts were made to develop new membranes of chemically and thermally stable polymers. Since 1990 the ion beams from the U-400 cyclotron have become available for the treatment of polymers. The scheme of the irradiation facility installed at the U-400 is shown in Fig. 5 [47].

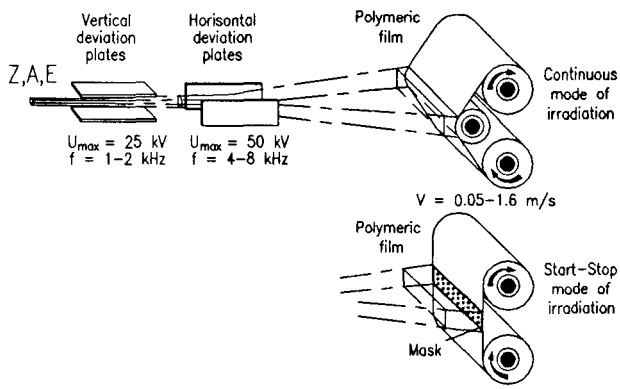

Fig. 5: Plan of the irradiation facility using a scanning beam from the U-400 cyclotron at FLNR JINR.

There are two modes of irradiation: a continuous one and a start-stop regime. The continuos mode with the film movement speeds from 0.05 to ca. 2 m/s is normally used for homogeneous irradiation of polymeric films with the thicknesses from 3 to 100 μm and the width up to 35 cm. The irradiation set up is provided with electrostatic and magnetic systems for the horizontal and vertical scanning, with gauges measuring the ion flux distribution within the irradiation zone, and with semiconductor detectors for the ion energy measurement.

The track membranes are known as precise porous films with a very narrow pore size distribution. The

homogeneity of the pore structure depends first of all on the track etch rate to bulk etch rate ratio which, in turn, depends on the bombarding particles. Fig. 6 shows the experimental data on the etch rate ratio in PET as a function of the parameters of projectiles. The etch rate ratio increases dramatically with increasing atomic number and flattens out at very high energy losses. That is why the best ions for creating microporous structure in polymers are rather heavy ones - for instance Xe and Kr in the case of PET. This tendency is valid for many polymers but the slope of the response curve can be very different for different plastics.

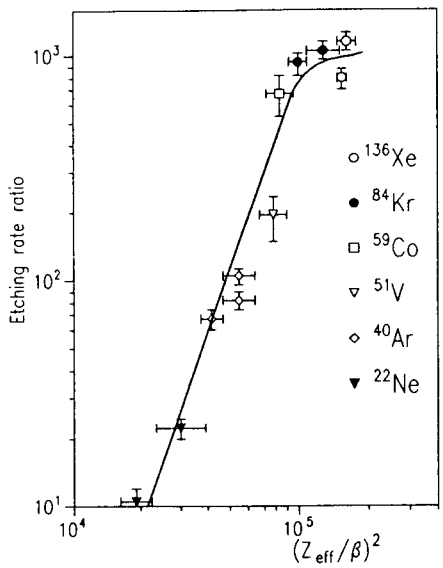

Fig. 6: Track etch rate to bulk etch rate ratio as a function of the bombarding particle parameter [8] (here Z_{eff} is effective charge of an ion and β is its relative velocity).

The initial material for the TM production is biaxially oriented PET of high quality, usually the film thickness is 10 μm. Recently the manufacturing of membranes with the thickness of 20 μm film has become available. The ion-irradiated films are subjected to ultraviolet sensitization and chemical etching [47,48]. The etching machine provides a continuous treatment of the film with the speed of up to 100 m/h. The finished membrane is subjected to a rigorous testing by electron microscopy, gas and water flow rate methods, bubble point measurements, and some others. The production potentialities are determined by the facilities available: the U-400 cyclotron - for the heavy ion irradiation, six machines for the UV treatment and four machines for the chemical etching. Up to 100 000 square meters of polymeric foils a year can be irradiated and chemically treated. The pore diameter in the produced PET TMs is ranged from 0.02 to 10 μm.

Track membranes of other polymers (polypropylene, polyvinylidenefluoride) are produced on the laboratory scale. The manufacturing procedure for these chemically resistant membranes differs from that for the PET TMs by the methods of sensitization and etching.

Track membranes find wide use which is predetermined by their special properties: definite pore size, small thickness, definite threshold of particle retention, absence of leachables. Most applications were considered in numerous papers (see [10,11,40,47,48]). At present, the membranes produced at FLNR are used mainly in biology, medicine and environment protection [49].

7.2 Modification of polymeric materials

In contrast to the track membrane production, other modifications of polymeric materials with cyclotron beams are at the more or less early stages of development. Let us outline some of them (see for details [5,6,50-53]):

- controllable change of the electric conductivity,
- controllable change of optical properties,
- waveguiding structures in polymers by ion implantation,
- creation of anisotropic copolymers by grafting into tracks,
- controllable change of the ferro ↔ paraelectric phase transition parameters,
- doping of the polymers by using latent tracks with the prospects to produce materials for nanoelectronics,
- fabrication of arrays of metallic or conductive polymer wires embedded into insulating polymer and creating materials with special magnetic and electric properties,
- adhesion improvement.

Conclusion

The present review can not be exhaustive; the interested reader should take into account that many relevant results obtained with ion implanters were not considered here. Some common features of high-LET effects in polymers can be successfully investigated by using ion beams of lower energies [54,55]. At the same time one should be careful by applying the information gained from experiments with implanters to the specific energy region of 1 to 100 MeV/u because nuclear collisions may have a strong influence on the radiolytic processes at lower energies [54,56]. Again, the studies performed with accelerators other than cyclotrons (for instance, UNILAC at GSI, Darmstadt) but providing valuable results in the field under discussion were out of consideration. Nevertheless, the author hopes that the history and the present status of polymeric materials research with cyclotrons are covered by this report more or less in balance.

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