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Historical Prolegomena

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1.1. A short history of sources of X-rays

As the story goes, on November 8, 1895, Wilhelm Conrad Röntgen stayed on at his laboratory late into the evening because he was intrigued by the observations he had just made. He was working on what was known as cathode rays using a Hittorf discharge tube. He was attempting to create total darkness in the room in order to detect the visible radiation (fluorescence), emitted by his device, using a fluorescent screen of barium platinocyanide. To do so, he covered the tube with a black envelope, opaque to visible light. When he turned the tube on, he saw a glow that disappeared when he turned it off. He noticed that this glow came from the fluorescent screen. Surprised by this, he sought to understand fully, as a thorough experimentalist would do, and continued his investigations: the intensity of light increased when he moved the screen closer to the tube. He continued his work after dinner and noted that what seemed to come from the tube was able to pass through thick screens of paper or wood (several centimeters thick) but could be stopped by lead.

He worked hard for several weeks to try to find out what these “rays” were (*X-Strahlen* as he called them) while keeping his discovery secret. Shortly before Christmas, he informed his wife Bertha and took a radiograph of her hand. On December 28th, he sent a report on his investigations to the Würzburg Medical Physics Society, whose Chairman agreed to publish it, even though no oral presentation was made at the time. Röntgen presented his results on January 23, 1896 to a meeting of the Physics Society where he explained:

X-ray Radiation and Artificial Bragg Structures,
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> By chance, I found that the rays passed through black paper […]. I then used the instrument as a camera, and the experiment worked.

At the end of this meeting, Röntgen successfully made a radiograph of the hand of the anatomist Albert von Kölliker, who proposed to name these rays X- or Röntgen rays, and this suggestion was approved. However, it is of note that the scientific publication presenting the discovery of X-rays does not mention the fortuitous nature of his discovery.

The "official" discovery of X-rays is a good example of a type of serendipity: it is highly probable that the production of X-rays was observed prior to Röntgen's discovery, but that the observers were not truly aware that they had discovered a new type of ray. Sir William Crookes himself, the inventor of the eponymous X-ray tube, observed irradiated photographical plates that had been placed near his tube, as did the doctor Arthur W. Goospeed. The physicist Yvan Pulyui, using a tube of his own design, noted photographical plates that were also irradiated by the radiation from his tube. However, Röntgen, with his expertise as an experimentalist, is responsible for the scientific approach (verification, attempt as characterization, etc.) that led to what can be called a scientific discovery. Serendipity played a full role in this.

Very quickly after Röntgen's discovery, many scientists set to work to reproduce Röntgen's experiments and determine the characteristics of this radiation. In particular, we can cite the work of Jean Perrin who repeated several of Röntgen's experiments and also demonstrated that X-rays were capable of discharging charged electrical bodies and of ionizing (in modern language) gases and metals (the "metal effect", using his expression) (Perrin 1948, 1956). Léon Gouy, a close friend of Perrin, established that if these rays behaved as waves, their wavelength must be considerably shorter than 1/100 of the wavelength of green light. It was not until Max von Laue's work on crystalline diffraction was completed in 1912 that the wave-like nature of these X-rays was demonstrated definitively. The "tormented" history of the discovery of the nature of X-rays, their physical origins and the crystalline diffraction is described in detail in the published work of Authier (2013).

In terms of research, the schools of Cambridge and Edinburgh became the main centers for X-ray spectroscopy, with in particular the works of Henry Moseley who successfully recorded the emission spectra of a large proportion of the elements in the periodic table, before his tragic death in 1915. His works contributed considerably to the construction of the atomic model by Niels Bohr. A long period of interaction ensued between newly discovered mechanics and the experimental results in the field of X-ray. Therefore, the multiplets observed by Alfred Landé in the X-spectra of heavy atoms led Wolfgang Pauli and Ralph de Krönig to the electron spin hypothesis.

Concerning the production of X-rays, improvements were made progressively. Until the 1920s, the main source remained the Crookes tube, which is based on a discharge in a gaseous environment under a partial vacuum which deteriorates over time (pressure ranging between $7\ 10^{-4}$ and $5\ 10^{-5}$ Torr). The electrons that generate X-rays come from ionization of the residual gas molecules, and are excited by the action of continuous electrical voltage from a few kV to several hundreds of kV. This voltage is then generally generated by Rühmkorff coils. Tubes based on this mechanism are sometimes referred to as cold cathode tubes. In fact, the Crookes tube is not entirely suitable for producing X-rays because it has several disadvantages for this task:

– The vacuum is "imperfect", which damages propagation of the electrons that collide with the gas molecules.

– To produce hard X-rays, the electrical voltage between the cathodes must be increased, which creates electrical beakdown issues.

To mitigate these problems, the solution consisted of creating a high vacuum (pressure lower than 10^{-6} Torr) within an airtight chamber, generally a glass bulb sealed after the pumping to create the vacuum. The electrons are produced by a thermo-ionic effect: a cathode that has been heated, generally directly by the Joule effect, emits, due to this thermal agitation, electrons that are then accelerated by high voltage (from a few kV to several hundreds of kV), as seen in Figure 1.1.

X-rays are produced when the atoms of a target known as an anode or sometimes anticathode[1] are bombarded with the electron beam, by means of two effects: *Bremsstrahlung* or braking radiation, and characteristic emission[2]. The first effect is due to deceleration of the electrons by the nuclei of the target's atoms. The intensity of the phenomena increases as the atomic number of these atoms increases, which explains why the anode is composed of heavy atoms, generally tungsten. The second effect is quantic in nature: it corresponds to the emission of energy in the form of electromagnetic radiation when an electron jumps from an electron shell to fill an electron hole; in the case of the X-ray tube, the ionizations are caused by the

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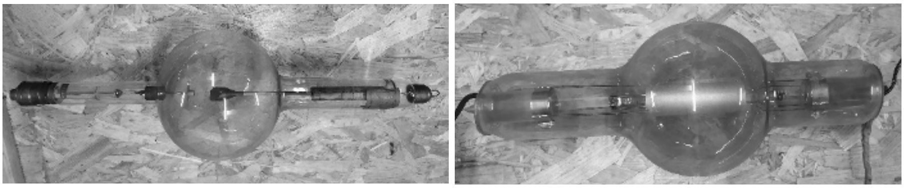
[1] The Crookes tube has three electrodes: a cathode and an anode between which the HV is applied, and a third electrode connected to the anode and positioned at an angle of approximately 45° to it. In the Coolidge tube, there are only two electrodes: an emissive cathode and a second (anode), sometimes also known as an anticathode, which is the target for "projectile" electrons and emits X-rays.

[2] The term X fluorescence also exists but the term is not correct because, in this case, the ionization must result from an irradiation by X-rays and not by electrons.

electrons, projectiles of the cathode beam. William D. Coolidge, who worked at General Electric, is responsible for fine-tuning the design of the eponymous tube in 1913 (see Figure 1.1), implementing the aforementioned principles. This type of tube was made in different forms in accordance with the works of several "inventors", generally doctors specializing in radiology. Even though it is more effective in terms of X-ray production than the Crookes tube, the efficiency of the Coolidge tube is only of the order of 1%. However, it remains the most frequently used source of radiation: classic radiology, scanners, tomography, spectroscopic analysis, etc. It is, or has been, made by several companies: General Electric (GE), Compagnie générale de radiologie (CGR), Philips, etc.



a) b)

Figure 1.1. *a) Coolidge X tube; b) kenotron (© P. Jonnard)*

We will mention that, while the X-ray tube itself was being manufactured, tubes known as kenotrons were also manufactured to rectify the alternating voltage and obtain the direct voltage required to create the HV or VHV field that generates acceleration of the projectile electrons. The kenotron is in fact a diode (valve) that can be mounted on a bridge rectifier to ensure full-wave rectification. Manufacturing of these requires the use of technologies close to the X-ray tube technologies themselves: vacuum technologies, glasswork, etc.

For specific applications requiring very hard X-rays, in other words with a photon energy nearing MeV, the source of "projectile" electrons can be made up of an electron accelerator, generally known as a LINAC (linear accelerator), or a "betatron" circular accelerator. Use of a Van de Graaff or the Cockroft–Walton electrostatic generator remains anecdotal.

In the field of research, the situation changed in the 1960s. Everything began with the observation of radiation known as synchrotron radiation. It is electromagnetic radiation with a spectrum ranging in practice from infrared to hard

X-ray[3], emitted by charged particles (electrons, or positrons) accelerated to a speed close to the speed of light, when they are subject to a centripetal force resulting from the action of a magnetic field. This phenomenon occurs in accelerators known as synchrotrons and storage rings. Initially, it can be considered a parasite effect for this type of machine, since it causes a loss of energy of orbiting particles that must be compensated for (Iwanenko and Pomeranchuk 1944). This radiation was also named orbital radiation.

Although the concept of synchrotron emission appears in various scientific works at the end of the 19th century (Liénard 1898), observation of it dates back to the 1940s: indirectly by the variation of particle orbits in 1946 by John Blewett and directly in 1947 by the observation of visible light on a 70 MeV synchrotron by a GE team. Driven by eminent scientists including W. Coolidge, the father of the X-ray tube, different circular accelerators were constructed in the United States, including the 70 MeV synchrotron at GE's laboratory in Schenectady. One of the problems occurring with this type of machine was intermittent sparking. Attempts were made to locate this visually in the machine using a mirror through a port that opened onto the interior of the machine. During an inspection of this type, on April 24th, 1947, a technician known as Floyd Haber saw a spot of light coming from one of the tubes. Having already eliminated the Cherenkov effect as the cause of this emission, physicists including Robert Langmuir and Herbert Pollock came to the conclusion that it was Schwinger radiation[4] (Pollock 1983). The circumstances of this observation caused a certain degree of controversy, and the pre-eminence of the discovery was contested on several occasions, in particular by George C. Baldwin (Baldwin and Kerst 1975). Be that as it may, this synchrotron radiation was considered by the "machine engineers" either as a problem, or as a way of characterizing their apparatus.

It was not until the 1960s–1970s that physicists specializing in X-ray spectroscopy became interested in synchrotron radiation as a potential source of X-rays. Professor Yvette Cauchois (see Figure 1.2), who at the time was director of the Chemistry Physics Laboratory (LCP) at the University of Paris, and a group of Italian scientists including Ugo Fano played a key role[5] in this area. Fano, who worked with Enrico Fermi and discovered the eponymous effect, had to immigrate to the United States

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[3] The spectrum range depends on the energy of the accelerated particles. The higher this energy, the more "hard" components it presents, in other words photons with higher energy levels (ultraviolet, x-ray).

[4] Julian Schwinger, an American theoretician and physicist, performed a theoretical study of synchrotron radiation, first in 1946 then in 1949 (Schwinger 1949).

[5] From 1927 onwards, Fermi set up a group of talented theoreticians including Ugo Fano, Ettore Majorano and Gulio Racca who focused on the application of the new quantum mechanics with regard to X-ray spectroscopy.

where he ended up at the National Office of Standards, the forefather of the prestigious National Institute of Standards and Technology (NIST).

Figure 1.2. *Yvette Cauchois in front of an X-ray machine (©Laboratoire de Chimie Physique - Matière et Rayonnement)*

Cauchois, aware of the interest of synchrotron radiation for X spectroscopy, met Fano who shared the same point of view. A fruitful working partnership ensued, named *Sanita Luce*, between the French and Italian teams following communications between Cauchois and Mario Ageno, who was at the time director of the physics laboratories of the Italian National Health Institute. The Italians had already worked on characterization of the X-ray emitted by the ADONE storage ring in Frascati, whereas the LCP had great expertise in X-ray spectrometers. The particle physics communities, Frascati’s Italian one and the French at Orsay, had previously worked together, in particular during the transfer of the Italian ADA accelerator to LINAC in Orsay. Cauchois created a team of young experimentalist researchers, Pierre Jaeglé, Pierre Dhez, Christiane Bonnelle, Robert Barchewitz, and technicians, including Henri Ostrowiecki; among the Italians, there were G. Missoni and G. Cremonese. Concerning hard X-rays, the LCP had a Cauchois spectrometer, operating by transmission, and a Johann spectrometer, operating by reflection under vacuum. Bonnelle was responsible for these crystal spectrometers; concerning soft radiation, a grating spectrometer had been developed by Jaeglé and Dhez in the new section of the LCP installed at Orsay. This group of scientists and technicians made up the pioneering team in X spectroscopy using synchrotron radiation.

It appears that the first spectrum obtained was an aluminum transmission spectrum in the vicinity of the K absorption threshold (see Figure 1.3). The conditions of this first result were described by Ostrowiecki (2014). The priority sometimes claimed for this spectrum obtained using synchrotron radiation may be debated insofar as D.H. Tomboulian and P.L. Hartman, in the mid-1950s, characterized the radiation emitted by the 300 MeV Cornell synchrotron using a grating spectrograph and the Be K and Al L thresholds (in the field known as Vacuum UltraViolet (VUV) and not in the field of X-ray) had been observed (Tomboulian and Hartman 1956). Be that as it may, following this initial success obtained from the French–Italian collaboration, a series of publications on the results of X-ray spectroscopy (Cauchois et al. 1963; Jaeglé and Missoni 1966; Jaeglé et al. 1967) and X-ray optics (Barchewitz et al. 1967, 1969) ensued. The works of Jaeglé and Dhez in Frascati were in the so-called Holweck field, to observe the emission lines of strongly ionized atoms in order to detect possible population inversions that would potentially lead to a laser effect in this domain. This turned out to be the case, and Jaeglé is considered the father of X-ray lasers obtained using hot plasma.

After a change of direction by the Italians, the working relationship ceased in 1971. The Italians recycled the Frascati synchrotron into an optimized source of synchrotron radiation and continued their works in the field of XUV and of soft X radiation (10–200 eV), with in particular Antonio Biancini in the Soliditi Roma group of spectroscopists. In France, Cauchois contacted scientific decision-makers to promote synchrotron radiation. In 1963, she contacted André Blanc-Lapierre to obtain assurance from the Laboratoire de l'Accélérateur Linéaire (LAL), which was in charge of constructing the ACO collider ring for particle physics studies, that a magnet with a strong magnetic field would be included for the purpose of producing hard X-rays; the forefather of the wiggler. This vision, far in advance of its time, unfortunately did not come to fruition for technical reasons and due to issues about sharing the machine with particle physicists, it would appear.

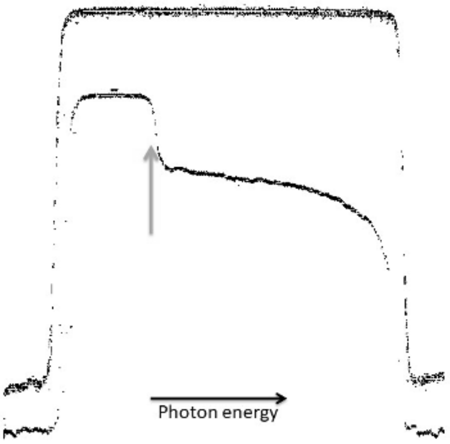

Figure 1.3. *Aluminum transmission spectrum obtained on the Frascati synchrotron on May 6, 1963 by a French–Italian team. The upper curve shows the continuous base obtained through a beryllium film; the lower curve shows the spectrum obtained through an aluminum screen. The green arrow indicates the discontinuity in K absorption of the aluminum. Figure adapted from Cauchois et al. (1963)*

In 1972, Cauchois brought the subject up again, now equipped with the results achieved at Frascati. Thanks to a well-constructed line project known as VUV, the cluster of young scientists (Yves Farge), including those who had returned from Frascati (Jaeglé, Dhez, etc.), and the support of influential scientists at the CNRS (Jacques Friedel and Hubert Curien), success was achieved! The considerable role played by the LCP in the use of synchrotron radiation in spectroscopy and X-optics is analyzed in the article by Bonnelle and Dhez (2014), and the history of this joint French–Italian effort is described in the article by Mottana and Marcelli (2013).

A laboratory called Laboratoire pour l'Utilisation du Rayonnement Electromagnétique (LURE), provided with funding and personnel, was created in 1971; in the spring of 1973, the line of light from ACO's magnet 8 was opened. Several lines of light, to employ the terminology used in synchrotrons, were then opened on the ACO, allowing original research on many themes. The physics and chemistry of atoms, molecules and of solids held an important position: atomic spectroscopy (photo-absorption) in the VUV field with, among others, Pierre Jaeglé, Pierre Dhez and François Wuilleumier, supported by theoreticians such as Michel Cukier and Françoise Combet-Farnoux (Cukier et al. 1974); X-ray photoelectron spectroscopy led by Jean Lecante's team; angle-resolved photoemission where Yves Petroff (who was director of the LURE then of the ESRF) and his team played a key role. This research led to many firsts (Thiry et al. 1979; Petroff and Thiry 1980):

– first complete band structure of a metal;

– illustration of the effects on N-bodies (in nickel);

– first observation of surface states on core states.

In addition, research in the field of X-ray optics using synchrotron radiation, which began with the working partnership between Robert Barchewitz and the Italian team, and was at the origin of firsts in what later became known as ReflXANES and ReflEXAFS (Barchewitz et al. 1978), continued with Jean-Michel André; this gave rise to the first determination of the continuous refraction index (real *and* imaginary parts) in anomalous diffusion regions in the field of X-rays (André et al. 1982). Experiments in the anomalous zones were then highly successful, in particular involving magnetic multilayers.

In 1975, ACO was abandoned by particle physicists and dedicated completely to the use of synchrotron radiation. The other collider on the Orsay campus, Dispositif de Collisions dans l'IGLOO[6] (DCI), opened up to the use of synchrotron radiation in June 1976 with cohabitation of the communities: particle physicists and synchrotron radiation users. The X-rays at this facility are harder than those available at ACO.

Research using synchrotron radiation was launched with first-generation equipment in various countries (Tantalus I in the United States, DESY in Hamburg, etc.). These would rapidly find themselves limited, and the race was on to create second-generation machines designed entirely to optimize the production of synchrotron radiation, obtain strong brilliance[7] and make researchers' work

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[6] IGLOO is the name given to the building that resembles, with its rounded shape, an igloo made from ice; it initially housed the targets room of the Laboratoire de l'Accélérateur Linéaire.

[7] Brillance is a quantity that qualifies the source; it is expressed in photons/s/mm^2/mrad^2/0.1% BW, where BW refers to the bandwidth.

"comfortable"[8]. With the experiments carried out on Tantalus I at the University of Wisconsin in the United States, the superiority of the electron storage rings as a source of synchrotron radiation was demonstrated. In a storage ring, the electron beam circulates continuously for hours, whereas in synchrotron machines, repeated injections are required. Sources of synchrotron radiation using storage rings were then constructed in the most industrialized countries: National Synchrotron Light Source (NSLS) at Brookhaven in the United States, Adalin at the University of Wisconsin (United States) succeeded Tantalus I, ALS at Berkeley (United States), the Photon Factory at the KEK laboratory in Japan, BESSY I in Berlin (Germany), Doris II and III in Hamburg (Germany), SSR in Daresbury (United Kingdom), Super-ACO in Orsay (France), MAX-I in Lund (Sweden) and Adone in Frascati (Italy).

Nevertheless, a whole swath of research was not yet accessible with this type of second-generation machine because the X-rays delivered were not hard enough, and due to insufficient brilliance. This is the case in particular for radio crystallography, with its significant application in the structural determination of molecules of biological interest. It became necessary to create so-called third-generation machines offering the required characteristics. Yet, the cost of these machines was becoming very high because delivering photons with greater energy requires larger machines and the particles in orbit in the machine have higher energy, as shown in Figure 1.4 showing the universal curve of the spectrum of synchrotron radiation produced by a curve magnet. It is characterized by the critical wavelength $\lambda_c(nm) = \frac{1.864}{B\,(E_p)^2}$, with B (in Tesla) being the magnetic field of the magnet and E_p (in GeV) being the energy of the particles in orbit in the machine. The smaller the value of λ_c, the more the spectrum tends towards hard radiation.

In addition, relatively costly so-called insertion elements (wiggler, undulator) must be included to increase the efficiency of radiation production. These elements installed on the straight sections of the machines are assemblies of N_d magnetic dipoles alternated on a regular basis, which impose a quasi-sinusoidal trajectory on the electrons. They are characterized by a parameter, K, which takes into account the period of the assembly and the intensity of the magnetic field: if K is less than 1, the insertion element is an undulator (K is typically 0.5) and the intensity emitted varies as a function of ${N_d}^2$following an interference phenomenon between the emissions at each dipole. On the contrary, if K is greater than 1, the element is a *wiggler* (K is typically 3) with emitted intensity varying as a function of N_d.

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[8] Working conditions for the first devices were sometimes difficult; in the ACO bunker, it was very hot in summer and difficult to stand up straight, with access only by ladder to top it all off!

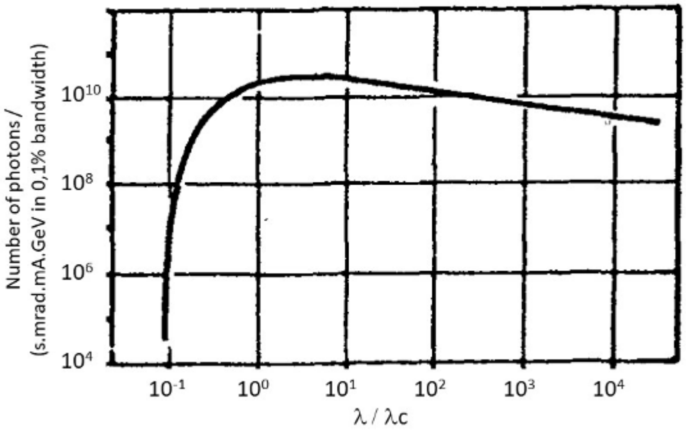

Figure 1.4. *Universal spectrum of synchrotron radiation for 1 GeV electrons as a function of the length of the scaled wavelength* $\frac{\lambda}{\lambda_c}$ *(λ_c is the critical wavelength). According to Bessière (1996)*[9]

The third-generation machines delivering soft X-rays were created at the national level (SOLEIL in France, Diamond in the United Kingdom, BESSY II, Petra III and ANKA in Germany, Elettra in Italy, SLS in Switzerland, Pohang Light Source in Korea, etc.) but for high energy machines (energy of an upper beam of 5 GeV) delivering very hard X radiation, collaboration between different European nations was required to build the European synchrotron ESRF, or investment by large countries capable of funding very large instruments (VLIs). This is the case for the Argonne National Laboratory (APS) in Chicago in the United States, PETRA at DESY in Hamburg and Spring-8 in Japan. Continuous improvements were made to most of these machines by introducing different techniques: continuous injection, slicing, etc.

In the field of X-ray production from an electron beam, an important step was made in the works of John Madey. In fact, in the 1950s, H. Motz at Oxford (United Kingdom) became interested in what is known as undulator radiation and proposed to use this device as a "light amplifier". His work was followed up by GE engineers who developed the *Ubitron* (Phillips 1960), consisting of a progressive wave tube where an interaction is established between an electron beam undulating under the

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[9] See doi: 10.1051/jp4:1996449.

effect of a magnetic field and an electrical transverse mode from a waveguide. We can refer to the forefather of the undulator used in storage rings to produce X-rays. These works were classified and “buried” until they were “exhumed” by J. Madey at the beginning of the 1970s. He succeeded, in collaboration with the High Energy Physics Laboratory team at the University of Stanford (United States) (Elias et al. 1976), in constructing a device based on the effect then known as the stimulated *Bremsstrahlung* (Madey 1971) that he named the Free Electron Laser (FEL).

Overall, the first FEL consisted of an undulator associated with an optical cavity used to confine the radiation within the undulator. The electrons were initially injected using a LINAC. However, Y. Petroff, having returned from the United States where he took part in a conference held by Madey, busied himself with installing an FEL device on the Super-ACO storage ring. Therefore, the first FEL operating on a ring was created; it was named the Centre pour Laser Infra-rouge d’Orsay (CLIO) because it emitted within the infrared spectrum by means of a mirror cavity. The presence of an optical cavity of this kind then restricted the FEL to the production of long wavelength radiation. A phenomenon known as self-amplified spontaneous emission (SASE) would then lead to the avoidance of use of the cavity and thus allow the wavelength to be shortened (Bonifacio et al. 1984). The SASE phenomenon is based on the use of noise (instability of the electrons) in a high gain nonlinear regime, causing saturation in a single pass, provided that the undulator is of sufficient length (Huang and Kim 2007). This operating mode, named SASE-FEL, is independent of the wavelength but given that it starts with a noise, it remains chaotic in nature, which manifests itself in instability on a spectral and temporal level and in the intensity of the radiation emitted. Figure 1.5 shows the principle of SASE-FEL.

The radiation is emitted in the form of a series of pulses with an envelope that has a temporal width of the order of tens to a few hundred femtoseconds and a spectral width of the order of tens of eV. Other than its “extraordinary” brilliance peak (10^{32} photons/s/mm^2/mrad^2/0.1% BW), this SASE-FEL radiation presents a transverse coherence due to the gain-guiding effect that synchrotron radiation does not offer. In order to improve the temporal coherence and reduce the bandwidth, different techniques have been proposed and tested or are currently being tested. This is mainly the seeding technique. In the field of extreme UV (EUV), seeding was performed using high-harmonic generation (HHG) emitted by a gas pumped by an infrared laser (Togashi et al. 2011). A wide variety of external input techniques have been developed: HGHG, EEHG, PEHG (Huang et al. 2021).

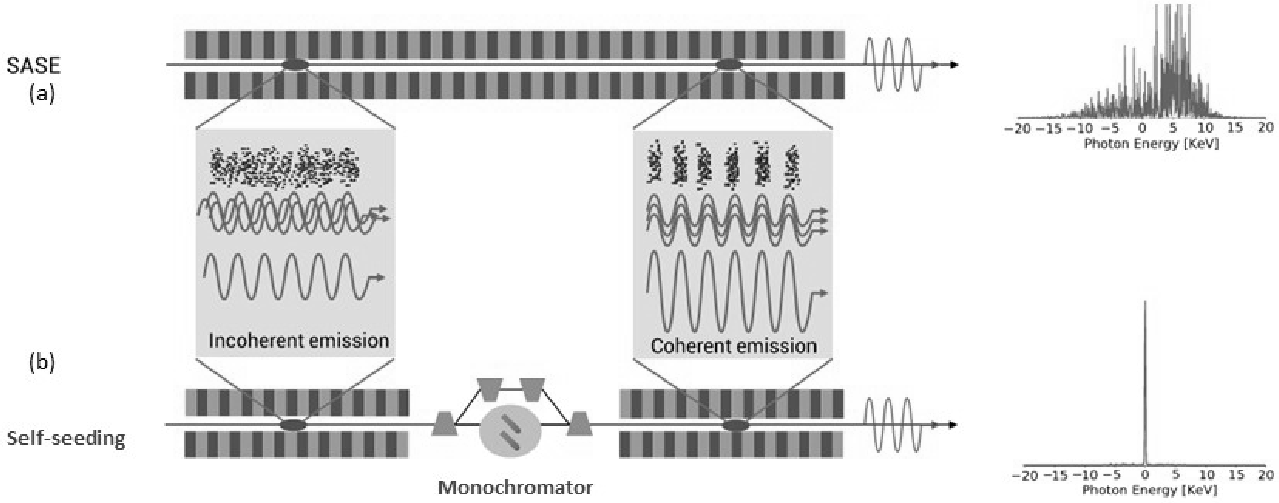

Figure 1.5. *Two operating modes of an FEL*

COMMENT ON FIGURE 1.5.– *a) SASE mode; b) self-seeding. At the entrance to the undulator (modulator), a spontaneous emission of radiation occurs in a linear regime with low gain; the radiation interacts little by little with the electrons which are in micro-packets and then emit coherently; the radiation follows a nonlinear regime with amplification and lastly saturation. In self-seeding mode, a monochromator is placed prior to amplification, which occurs in the radiator, to "purify" the radiation. Figure adapted from Huang et al. (2021)*[10]*.*

In the field of soft and hard X-rays, another technique known as self-seeding, which does not use an external source of radiation, was invented (Feldhaus et al. 1997). This consists of reinjecting filtered or monochromatized radiation into the SASE-FEL radiation using a Bragg monochromator (hard X-rays) or a diffraction grating (soft X-rays). Figure 1.5(b) shows the principle: (at least) two undulators are used. The first constitutes a so-called "modulator", which operates in SASE mode and in low gain regime, whereas the second, sometimes named a radiator, operates under a high gain regime, potentially to the point of saturation. The technique of self-seeding could benefit artificial Bragg structures in the XUV and soft X-ray fields, as we will demonstrate in Chapter 8. Research on self-seeding continues to be highly active in the various XFEL centers, in particular at the European XFEL (Geloni et al. 2012).

The performances of FEL in terms of brilliance (high intensity), temporal structure and coherence have already resulted in a multitude of results; among these, we can cite in particular:

– for brilliance: illustration of the self-amplified spontaneous emission and stimulated emission in the field of hard X-rays (Yoneda et al. 2015), soft X-rays (Rohringer et al. 2012; Beye et al. 2013) and EUV (Jonnard et al. 2017), intensity-induced X-ray transparency (Young et al. 2010; Stöhr and Scherz 2015), stimulated Raman emission (Weninger et al. 2013), saturable and two-photon absorption (Hoffmann et al. 2022);

– for the temporal structure and coherence: coherent diffractive imaging (Chapman et al. 2006), Fourier transform holography (Gorkhover et al. 2018).

Table 1.1 lists the main XFEL installations around the world, along with their characteristics. Figure 1.6 shows the historical evolution of sources of X-ray based on the use of electrons (or positrons) and of their brilliance. Eduard Prat's recent article

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[10] See doi: 10.1016/j.xinn.2021.100097; from the journal *The Innovation*, a Cell Press partner journal.

(Prat 2021) and the references included in it provide a more in-depth discussion of the principles of synchrotron radiation sources and free electron lasers.

Installation	Energy of the beam (GeV)	Energy of the photons (eV)	Rate of repetition (Hz)	Width at half-maximum of the pulse length (fs)
FLASH	0.35–1.25	14–620	4,000–10^6	10–200
LCLS	2.5–16.9	2,800–12,800	120	5–400
SACLA	5.1–8.5	4,000–20,000	60	2–10
FERMI	1–1.5	20–310	50	30–100
PAL-XFEL	3.5–10	275–20,000	60	5–100
SwissFEL	2.1–5.8	250–1,240	100	1–20
European XFEL	8.5–17.5	240–25,000	2.7×10^4	3–150
SXFEL	1.0–1.6	124–1,000	50	30–1,000
LCLC-II	4–15	200–25,000	120–10^6	1–500
SHINE	8	400–25,000	10^6	3–600

Table 1.1. *Performance data for the main XFEL facilities around the world*

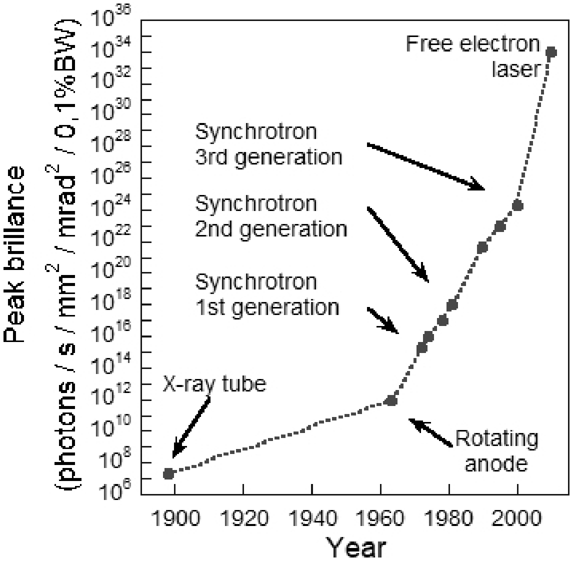

Figure 1.6. *Changes in sources of X-rays over time, based on the use of electrons (or positrons) and their brilliance*

Concerning the production of X-rays using relativistic electron beams, other techniques based on different phenomena can be implemented. We can cite inverse Compton scattering, channeling radiation, transition radiation and parametric radiation (Rulhusen et al. 1998). The latter two can benefit from artificial structures, as we will see in Chapter 8. However, we point out that to date, none of these techniques has resulted in facilities comparable to the synchrotron which delivers radiation on request for users, but that various projects for compact sources are in progress or being finalized. Therefore, we will mention the ThomX project in Orsay (France), targeting the production of radiation at 45 keV by Compton scattering (Bruni et al. 2016), the parametric X-ray (PXR) source project in Japan (Hyun et al. 2018) and X-ray source projects using channeling (Piot et al. 2012).

Aside from the production of X-rays from an electron beam, another technique has been put to good use: emission by highly ionized atoms generally obtained from hot plasmas[11]. An ionized atom is likely to emit characteristic radiation. The advantage of hot plasmas is that under certain conditions, it is possible to obtain a population inversion, as P. Jaeglé had predicted despite the criticism that he faced[12], with, as a consequence, the possibility of stimulated emission and ultimately laser emission. Jaeglé's group played a considerable role in the development of this type of laser, and currently a laboratory resulting from the works of his group, named LASERIX, and which is a platform of the University of Paris-Saclay (France), provides laser radiation in the XUV field; this platform offers the scientific and industrial community access to a wide range of coherent sources of radiation. Several other facilities operate around the world: the Compact Multipulse Terawatt (COMET) system at the Livermore National Laboratory (LLNL) in the United States, Prague Asterix Laser System (PALS) in Prague (Czech Republic). Development of this type of XUV laser has benefitted from multilayer artificial structures for the creation of the optical cavities that provide the retroaction. The groups led by Matthews et al. (1985) and Suckewer et al. (1985) played a pioneering role in this field.

Interaction of the laser beam with matter is also a source of short wavelength radiation. The characteristic of the radiation depends on the characteristics of the laser (power, spectral field, etc.). For medium power lasers emitting femtosecond pulses in the infrared spectrum, the phenomenon of HHG allows radiation to be produced with a spectrum up to the XUV domain. Laser radiation is focused on a jet of rare gas which uses a nonlinear jet to generate attosecond pulse trains (Drescher et al. 2001). This type of quasi-coherent source is frequently used in experimental studies of ultrarapid phenomena in atomic and molecular physics (Catoire et al. 2014). These sources benefit from Bragg multilayer optics.

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[11] A plasma is a state of matter composed of ions and electrons from ionized atoms.

[12] The probability of stimulated emission decreases as wavelength decreases.

Interaction of an ultra-intense TW (10^{12} W) or PW (10^{15} W) laser beam operating in a femtosecond regime with solid matter leads to the emission of radiation with very short wavelengths up to gamma rays. The electric field of the incident laser causes oscillation of the surface of the target at the laser's frequency with speeds ranging from zero to the speed of light, forming a so-called oscillating mirror. At its heart, the electrons vibrate at frequencies ranging from the laser's fundamental frequencies up to high harmonics reaching the X-ray domain. The Apollon laser, installed at the Saclay plateau (France), will allow this type of operation to take place and will soon be followed by extreme light infrastructure (ELI) European facilities (Amiranoff 2016).

These various radiation sources have allowed exploration of a large part of the spectral domain of X-rays. Depending on the physical phenomena studied or on the many technical means implemented to disperse and detect the radiation, the X-ray domain has been subdivided into several sub-domains (see Figure 1.7). The limits separating these various domains, which can be further divided, are arbitrary.

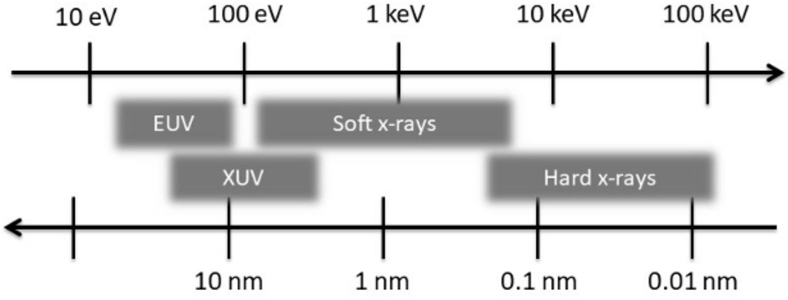

Figure 1.7. *The electromagnetic spectrum, with X-rays in the high energy or short wavelength domains, divided in turn into various sub-domains; EUV is the extreme ultraviolet domain*

1.2. A short history of artificial Bragg structures and changes in them

1.2.1. *Older work*

This book is about artificial Bragg structures and their application to X-rays. This type of structure resembles a photonic crystal, as it is now agreed that it should be called. Structures that we will be considering have periodic modulation of the optical index according to a spatial dimension and will therefore be unidimensional, corresponding to a one-dimensional photonic crystal (1D-PC).

The term photonic crystal, introduced in 1989 by Yablonovitch and Gmitter (1989), has its origins in the close analogy between the propagation of electrons in a crystal and the propagation of the photons in a (generally artificial) periodic optical structure. The terminology for photonic crystals has been analyzed by Yablonovitch in an article in which he insists on the need for a significant contrast in the optical index in order to characterize a photonic crystal. As for us, we will use the term "photonic crystal" for a periodically modulated optical environment in the spatial dimension and which creates strong coupling through this periodic arrangement with the electromagnetic radiation that propagates in it, in this case X-rays. In practice, this implies that the period involved is close to the wavelength of the radiation or to a multiple of it.

Long before this concept of photonic crystals, important figures in physics tackled the issue of propagation of an electronic or electromagnetic wave in a periodic structure. Illustrious names in the field of mathematics and physics between the 17th and 19th centuries can be associated with the propagation of a wave (acoustic, optical, etc.) within a one-dimensional network. Among these, we can cite Isaac Newton for the problem of the speed of propagation of sound; Jean Bernoulli and Daniel Bernoulli jointly for the principle of superposition; Augustin Cauchy and William Thomson (Lord Kelvin) for the study of wave dispersion; Joseph Fourier for the problem of heat propagation. Historically, it appears that John W.S. Rayleigh, in his works in 1887–1888 (Rayleigh 1887, 1888), following those of George G. Stokes, can be considered responsible for the discovery of the notion of a band gap, associated with an increase in reflectance with acknowledgment that the problem is mathematically connected to George W. Hill's equation.

In 1946, the publication of the famous work by Léon Brillouin, entitled *Wave Propagation in Periodic Structures* (Brillouin 1946), set down the academic foundations of theoretical physics concerning wave propagation in material environments with a spatially periodic structure. In it, the author presents diffraction of X-rays by crystals and the dynamic theory pioneered by Max von Laue, Paul P. Ewald, Charles G. Darwin, Walther Kossel, William H. Bragg and William L. Bragg (father and son) to mention just a few. Their works constitute the basis of modern physics of photonic crystals under the meaning we attribute to it.

In 1928, Bloch (1929) expanded on the results of Floquet (1883) to draw up a theory of propagation of electrons in crystals, which would be transferred to the propagation of photons in photonic crystals.

1.2.2. *Modern work*

In the 1960s, theoretical works concerning periodic structures related mainly to the exact resolution of the wave equations in sinusoidal or laminar environments, to spatial–temporal propagation in periodic environments and to radiation from sources included in such environments. From the late 1970s onwards, considerable technological innovations occurred, coupled with significant progress in modeling: active materials were introduced in connection with retro-distributed lasers, anisotropic materials, nonlinearity, magneto-optical, etc. All these contributions continue to form the substrate for the majority of contemporary works.

At the end of the 20th century, two fields developed at the cutting edge of the world of one-dimensional photonic crystals: the field of inferential mirrors at the nanometric scale for X-ray and XUV, which filled a gap in the optical systems in this spectral field with many applications in spectroscopy and imaging, and the field of fiber Bragg gratings which marked a considerable advance in the field of telecommunications. This book is at one of those frontiers, in other words the application to the X-ray domain.

The creation of reliable artificial Bragg structures for the short wavelength domain began at the end of the 1970s. In fact, the use of such structures was envisaged for X-rays from the 1930 onwards by von Deubner (1930) and then by DuMond and Youtz (1935). The structures created (using silver) were unstable (due to interdiffusion), and it was not possible to use them in practical applications. Following the attempts of Dinklage (1967) and then Croce and Pardo (1970), it was not until the mid-1970s, when technologies allowing films to be deposited at the nanometric scale were mastered, that a few groups created effective Bragg mirrors in the XUV domain that remain relatively stable over time despite interdiffusion issues.

Spiller (1972), benefitting from the resources of the company IBM, can be cited as a pioneer in the development of these structures. Several research and academic institutions in various countries (mainly the United States, Germany, France, USSR) then joined the race. Therefore, we can mention the works by the Germans, in particular at DESY (synchrotron radiation center in Hamburg) (Haelbich and Kunz 1976), the works of a group of French laboratories led by Dhez and lastly the works of various Soviet laboratories (Gaponov et al. 1983).

Following an initial period of research and development, several private companies manufactured and marketed interferential X-ray mirrors, mainly in the United States (Ovonyx, etc.) and in Europe (LEP, Xenox, Incoatec, etc.), whereas institutional laboratories continued to manufacture structures generally for research purposes, sometimes outsourced and often at synchrotron radiation centers. Among these, we can note Troy Barbee's group in the United States, a group at the Institute of Theoretical and Applied Optics in France directed by Jean-Pierre Chauvineau and then Franck Delmotte, who worked with NASA for the solar telescope project SOHO, and the "Optics" group at the ESRF European synchrotron with Éric Ziegler and Andreas Freund.

At the same time, characterization devices were developed at synchrotron radiation centers; these were reflectometers operating in the field of soft X-ray: MOGOTOX at Super-ACO (Barchewitz and Marmoret 1988) followed by the metrology center installed at the SOLEIL synchrotron (Idir et al. 2006) in France, the Reflectometry Station at the Helmholtz Zentrum Berlin (Sokolov et al. 2014) and the metrology devices at the PTB laboratory installed at the BESSY II synchrotron in Germany, the BEAR station at the Elettra synchrotron in Italy (Nannarone et al. 2004), the CXRO reflectometer installed at the ALS synchrotron in the United States, etc. Other reflectometers installed in academic laboratories have been developed with sources of different kinds such as MONOX (André et al. 2005) or CEMOX (Hecquet et al. 2006).

Aside from these reflectometers, reflectometry operating with grazing incidence and generally using commercial equipment, has been widely used to characterize the type and quality of the interfaces between layers. Other techniques such as high-resolution X-spectrometry and mass spectrometry (Galtayries et al. 2010) or electronic microscopy (Häussler et al. 2007) have also been developed and have largely contributed to the understanding of the physical and chemical processes involved in creating these Bragg structures. Another important step forward in the field of artificial Bragg structures was obtained when different groups began to combine micro-manufacturing techniques used in microelectronics with manufacturing techniques used for Bragg X-ray mirrors.

From around the mid-1980s onwards, a group with Vitaly Aristov and Alexei Erko installed at Chernogolovka near Moscow (USSR) began to manufacture lenses known as Bragg–Fresnel lenses (Aristov et al. 1988), while in France at the LCP, Barchewitz and J.-M. André initiated manufacturing of lamellar multilayer gratings in Bragg mirrors (Barchewitz and André 1996). The purpose of the Bragg–Fresnel lenses was to focus the X-ray, whereas lamellar multilayer gratings allowed "high" spectral resolution Bragg monochromators and polychromators to be created. We will develop this subject in Chapter 7.

Since then, technologies have advanced unabated, enabling the deposition of nanometric thin layers on flat or curved optics, of high quality in terms of surfacing and polishing, to obtain a broad array of devices: mirrors, monochromators or lenses, intended for synchrotron light lines, for astrophysics, for X-ray microscopy, etc. The use of sliced multilayer structures at nanometric dimensions operating in transmission mode (Laue mode) has led to the creation of focusing lenses, with spatial resolution of the order of a few nanometers.

Aside from applications in optics, microscopy and spectroscopy, specific physical phenomena generated within Bragg structures have been discovered: the Purcell effect, Kossel diffraction, Bragg–Raman diffusion or topological effects. In addition, periodic multilayer structures have also been proposed and tested as elements, allowing X-rays to be generated according to certain principles: resonant transition radiation, parametric radiation, free electron laser, retro-distributed laser, etc. These various subjects will be discussed in Chapter 7.

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