

INSTRUMENTS AND METHODS OF INVESTIGATION

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Low-power X-ray tubes (the current state)

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Contents

Abstract. **The operation principles and designs of X-ray tubes are discussed. Historical highlights and the current status of X-ray technology are reviewed and prospects for improving the technology are outlined. A comparison between the properties of X-ray tubes and those of hot cathodes and field-emission cathodes is given.**

1. Introduction. Discovery of X-rays

The first paper on X-rays was the report entitled "On a new kind of rays", published by Wilhelm Conrad Röntgen in the journal of the Wurzburg Physico-Medical Society on 28 December 1895 (see also Ref. [1]). Röntgen himself objected to giving his name to the radiation discovered. That is why in many languages it has come to be known as X-rays – the name given by Röntgen. The cathode-ray tube which Röntgen used in his experiments had been developed by J Hittorf and W Crookes (Fig. 1). The operation of this tube gives rise to X-rays, as was shown in Crookes's experiments, and from 1892 in experiments by Heinrich Hertz and his pupil Philipp Lenard. However, none of them realized the significance of their discovery and none published their findings.

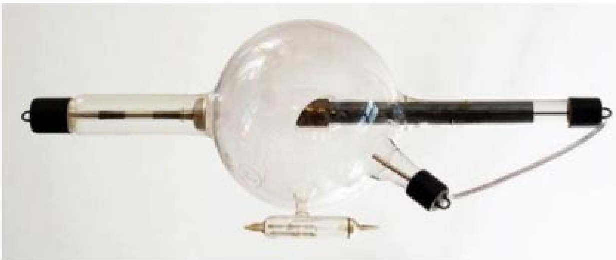

Figure 1. Crookes tube.

For this reason, Röntgen did not know about the prior discoveries and discovered the rays independently — in the observation of fluorescence emerging in the cathode-ray tube operation. When an electric discharge was passed through this tube, Röntgen observed a phenomenon which he described in the following way: "A cardboard screen painted with barium platinicyanide, when it was approached to the tube with a light-tight black cardboard covering, shimmered at every discharge initiated in the tube — began to phosphoresce. The simplest assumption about this phenomenon is that the black cardboard, which is opaque to visible and ultraviolet solar rays as well as to the rays of an electric arc, is penetrated by some agent, which gives rise to intensive phosphorescence...." In a series of experiments, Röntgen determined that this 'agent', which he termed X-rays, also passed through other bodies opaque to ordinary light: paper, wood, ebonite, the human body, and metallic layers. Röntgen also ascertained that low-density materials, which consist of light atoms, are more transparent than higher-density ones. In particular, a lead plate keeps out X-rays much more than an aluminum plate of the same thickness.

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Röntgen occupied himself with X-rays for a little more than a year (from 8 November 1895 through March 1897) and published three papers about them to give an exhaustive description of the new rays. Afterwards, the hundreds of papers by his followers published over a period of 12 years could neither make significant contributions nor make significant changes. Röntgen, who lost interest in X-rays, would tell his colleagues: "I have already written everything, do not waste time." In 1905, Röntgen was awarded the first Nobel Prize in Physics "in recognition of the extraordinary service he has rendered by the discovery of the remarkable rays subsequently named after him", and in so doing the Nobel Committee emphasized the practical importance of his discovery.

Over the past years, X-ray techniques of investigation, which rely on the unique properties of X-rays and the diversity of X-ray–matter interactions, have come into being and occupy a prominent place in science.

At the present time, X-ray techniques are employed to investigate the chemical composition of substances, the structure of crystalline materials and the effect of ambient factors on their properties, and the dynamics of fast processes in opaque media [2, 3], to measure small heights and distances, the thickness of coatings and sheet rolled bars in the course of manufacture, and the density of materials [4–6], to nondestructively inspect the quality of industrial products, to diagnose the quality of seeds [7–9], and so forth. X-rays are used to modify the properties of polymer materials [10], and X-ray lithography techniques in microelectronics permit making submicrometer-sized structures [11].

X-ray methods have occupied an important place in medicine: general diagnostics, the inspection of individual organs, traumatology, stomatology, therapy, etc. [6, 12]. Despite the emergence of new nonradiative diagnostic techniques reliant, in particular, on the use of ultrasound and nuclear magnetic resonance, the sales volume of X-ray equipment continues to increase, not decrease.

2. Operation principle of an X-ray tube

The wide use and development of X-ray methods calls for continual improvement in the corresponding X-ray equipment and, in particular, its main element — X-ray tubes [13].

X-rays emerge in the strong acceleration of charged particles (bremsstrahlung) or in high-energy transitions in inner atomic electron shells (characteristic radiation). Both effects are employed in X-ray tubes. The basic structural elements of such tubes are a metallic cathode and anode (also called an anticathode earlier). The electrons emitted by the cathode in X-ray tubes are accelerated under the action of the electric potential difference between the anode and the cathode (no X-rays are emitted in this case, because the acceleration is too weak) and impinge on the anode to undergo a sharp deceleration (Fig. 2). Two mechanisms are responsible for the generation of radiation in the X-ray range: one of them is associated with the bremsstrahlung effect, and the other is due to the knocking-out of electrons from the inner electron shells of anode atoms. The vacancies in the shells are filled by other atomic electrons. Emitted in this case is X-ray radiation with the energy spectrum characteristic of the anode material.

For electron sources in modern X-ray tubes, use is made of thermoemission (heated) and field-emission (cold) cathodes.

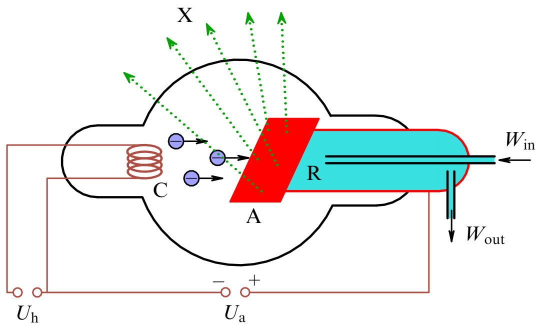

Figure 2. Schematic of an X-ray tube: X — X-rays, C — cathode, A — anode (sometimes termed "anticathode"), R — heat removal, U_h — cathode heating voltage, U_a — accelerating voltage, W_{in} — water cooling inlet, and W_{out} — water cooling outlet.

The thermoemission cathode of an X-ray tube comprises usually a spiral or a straight tungsten filament incandesced by electric current. By adjusting current in the filament circuit of the X-ray tube, and hence its temperature, it is possible to change the number of electrons emitted by the cathode. For a low voltage, not all electrons emitted by the cathode participate in anode current production, and a so-called electron cloud forms at the cathode. With increasing voltage, the electron cloud disperses and, beginning with a certain voltage (the saturation voltage), all electrons reach the anode. In this case, the tube carries a current at its maximum (the saturation current).

Along with common requirements for the cathodes of electric vacuum devices (to provide the requisite and stable emission current throughout the service life, to be well degassible and not to impair vacuum in the device in the operating regime, to have a sufficiently long service life, etc.), several special requirements are also imposed on the hot cathodes of the tubes, such as the operation stability for a high field strength at the cathode surface and the possibility of adjusting the cathode temperature (the emission current) over a wide range. Considering that the power of X-ray tubes is primarily limited by the anode thermal regime rather than the cathode current density, for electron sources use is usually made of hot cathodes of pure or carbidized thoriated tungsten.

The working temperatures of a tungsten cathode lie in the 2300–2650 K range, providing the emission current density that may amount to 0.3–0.7 A cm^{-2} for an efficiency of 2–10 mA W^{-1}.

In several X-ray tubes (for instance, in tubes for structure analysis, where it is important to rule out the ingress of the cathode material to employ target due to thermal evaporation), it is necessary to employ cathodes with a lower working temperature. Carbidized thoriated tungsten cathodes are employed in this case. The working temperature of these cathodes amounts to 1900–2000 K, the current density to 1–3 A cm^{-2}, and the efficiency to 50–70 mA W^{-1} [14].

In cold-cathode X-ray tubes, free electrons are generated under the action of a very strong electric field at the cathode surface (the field emission). The electrons emitted by the cathode are accelerated under the action of anode–cathode electron potential difference and impinge on the anode to undergo a sharp deceleration.

The cathode shape and dimensions largely determine the most important X-ray tube parameters — the shape and dimensions of the actual focal spot, which is used in reference

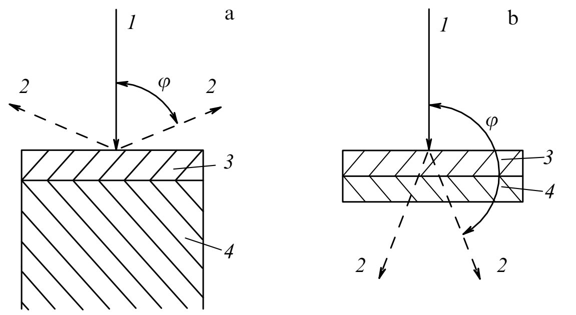

Figure 3. Massive (a) and transmission (b) anode designs: *1* — electron beam, *2* — X-ray radiation, *3* — target, and *4* — anode body (substrate).

to the segment of the target surface bombarded by the electron beam.

The anode of an X-ray tube designates the electrode which serves as the target or bears the tube's target. In investigations of the influence of anode material on the emission of continuum radiation, it turned out that the total radiation energy of the tube was directly proportional to the atomic weight of the element of the anode material for the same voltage and current in the tube. Subsequent investigations showed that the total radiation energy is proportional to the atomic number rather than the atomic weight of the element. This fact was ascertained by measurements of the total energy of the radiation from the anode. The part of X-ray radiation (emerging in the deceleration of electrons at the target) intended for an efficient use and confined in the solid angle whose vertex lies at the center of the actual focal spot is called the working beam of tube radiation. The geometrical characteristics of the working radiation beam (its direction and solid angle) depend on the design of the X-ray tube and its anode.

Structurally, the massive and transmission (shoot-through) anodes are recognized (Fig. 3). With the help of a massive anode (Fig. 3a), the working beam can be formed in the limits from $0° - 87°$. Such an anode consists, as a rule, of an anode body (*4*) and a target (*3*) (compound anode). The material of the anode body must possess a high thermal conductivity, because heat is removed to the cooler through the anode body. The anode body is most often made of copper, which possesses a sufficiently high melting temperature (1360 K), good vacuum properties, a high specific heat, and a high thermal conductivity.

The target is required to have a high melting temperature and a low vapor density at a high temperature. In the tubes intended for obtaining bremsstrahlung, targets are made of tungsten with a melting temperature of 3650 K. To induce the characteristic radiation of specific hardness, various materials (chromium, iron, copper, molybdenum, silver, etc.) may be utilized for a target. In some cases, the target as a structural element is missing from the tube, and its function is fulfilled by the anode body surface (a heterogeneous anode).

Transmission anodes are employed in X-ray tubes, as a rule, when it is required to produce a working beam whose axis and direction coincide with those of the electron beam (Fig. 3b).

3. X-ray sources

Microfocus radiation sources made their appearance in domestic X-ray engineering in the 1950s [15]. The main

Figure 4. BS-1 X-ray tube.

requirements specified to their design were a small focal spot and a short focal distance. Fulfillment of these conditions permitted placing an object under control near the focal spot to obtain sharp images of the object at a high magnification, in accordance with the direct-magnification recording scheme.

However, the practical samples of such radiation sources were heavy, bulky, and complex in operation. More convenient in application and simpler in design are radiation sources based on sealed-off microfocal X-ray tubes with an offset anode [16]. The first domestic electric vacuum device of this type was a 50-kV BS-1 X-ray tube (Fig. 4) manufactured by the Svetlana Special Design Office (Leningrad). The design of this tube, which determined for decades the main line of development of microfocal X-ray sources in our country, may be regarded as classical.

The tube comprises a 35-mm long anode tube 8 mm in diameter, at the end of which is sited a shoot-through type target. An annular permanent magnet is located on the anode tube, which focuses the beam preformed by a three-electrode gun. A power of 2.5 W for a focal spot diameter of about 40 μm provides an X-ray intensity sufficient for a variety of technological as well as medical applications [17]. The indicated design features of the BS-1 tube are inherent in all devices of the BS series developed in subsequent years, for instance, the 100-kV BS-6 and 150-kV BS-15 modifications.

Based on these tubes, a number of small-sized microfocus X-ray sources (RI series) were developed for a maximum voltage ranging 50–150 kV. The radiation sources of the RI series were designed using a single-block scheme, unlike the radiation sources with a constant pumping, which are made, as is well known, according to a cable scheme [18]. In a cable scheme, a high voltage is applied to the X-ray tube via a high-voltage cable from a generator. Consequently, a cable type radiation source contains three functionally separate high-voltage units, each of which — the generator, the cable, and the radiator with the tube — comprises special structural elements and connectors ensuring the high-voltage insulation in interunit connections.

Siemens GmbH (Germany) manufactures STRATON X-ray tubes for medical tomographs, which are distinguished for high-speed imaging and high image quality. They make use of direct oil cooling of the anode. A compact design allows accommodating two tubes in one system. These special features permit eliminating motion artifacts. Furthermore, there is no delay in the overheating, even for long scans.

Comet (Switzerland) and Philips (Netherlands) produce hot-cathode X-ray tubes with accelerating voltages from 9 to 100 kV for medical equipment. These tubes are widely employed for roentgenoscopy and in X-ray tomographs.

X-ray tubes from Oxford Instruments (Great Britain) are suited for industrial or scientific equipment and are characterized by a high stability, reliability, and low cost (Fig. 5). Different materials are employed as targets; the anode voltage

Figure 5. X-ray tubes from Oxford Instruments.

Figure 6. X-ray tube with a field-emission cathode from Oxford Instruments.

ranges from 4 to 80 kV, and the focal spot size is equal to 10 μm.

Furthermore, Oxford Instruments is engaged in the design of new X-ray tubes, including those containing field-emission cathodes (Fig. 6). Sarrazin et al. [19] proposed the utilization of carbon nanotubes as the cathode material, which permits decreasing the size and weight of the tube, as well as improving its efficiency.

Toshiba became the first Japanese company which engaged in X-ray tube production in 1915. Since then, their tubes are characterized by a high quality and reliability. The line of Toshiba products spans the entire spectrum of X-ray equipment applications in medicine, dentistry, and industry.

Finally, Varian Medical Systems, Inc. (USA) develops and manufactures X-ray tubes for several areas of the industry, including products for security applications, scanning with the aid of computer tomography, X-ray radiography, and other scientific applications. The X-ray tubes may be optionally enclosed in a glass or ceramic shell. Varian Medical Systems also offers bifocal X-ray tubes.

4. Fields of application of X-ray sources

X-ray radiation enjoys wide application in different fields of science, technology, and medicine. The high penetrating power of X-rays permits obtaining information about the internal structure and state of substances and living organisms. The latter is especially widely used for the purposes of medical diagnostics and treatment.

In a radiographic examination, X-ray quanta are transmitted through a substance and the radiation transmitted through the object is recorded. In this conditions, different photon–substance interaction mechanisms may be triggered. For a relatively low energy of X-ray photons, the main process determining the interaction is photoelectric absorption. As a result, an incident quantum is absorbed by an atomic electron in the shell with the ejection of a free electron with an energy comparable with the energy of the absorbed quantum [20].

Furthermore, other processes may occur in the passage of X-ray radiation through a substance, when the electron knocked out of the atom acquires only a fraction of the incident photon energy. This electron is termed the recoil electron. The remaining energy is radiated in the form of a quantum with a lower energy than the incident photon energy (Compton scattering). The direction of emission of the resultant X-ray quantum is arbitrary.

Since Compton scattering takes place on the atomic electrons of light elements, which the human body is primarily composed of, in a radiographic examination it comprises an appreciable part of the radiation transmitted through the patient's body and is responsible for the image blurring of the object under examination. To weaken this effect, a secondary radiation grid is employed in medical examinations [21]. However, the transmitted radiation intensity is lowered several-fold in this case, and the patient's irradiation dose has to be increased.

The influence of Compton scattering is substantially reduced when use is made of scanning radiography techniques. In this case, the object is exposed to a narrow X-ray beam, which is many times narrower than the features of the object under examination [22, 23]. As a result, the influence of Compton scattering from the neighboring domains on the object's domain of interest becomes insignificant, because these domains are not irradiated at the instant of recording.

The scanning recording technique permits obtaining a high image quality using low-dose X-ray irradiation. With KARS photofluorographs [24], for instance, a well-defined structure of the vertebral column is observed against the background of a high-quality image of a lung tissue pattern for a dose of about 0.05 mSv per radiogram (Fig. 7).

Modern X-ray tomographs permit performing all kinds of tomographic examinations known in the world. They may be employed for examining the brain, chest, abdominal cavity, etc. For instance, a Toshiba Aquilion Multi 4 instrument has a protocol for heart examination with a retrospective gating. This makes it possible to examine cardiac blood vessels, perform postoperative bypass angiography, and reveal the existence of microcalcium regions in coronary vessels.

Siemens produces a broad spectrum of equipment for computer tomography, both stationary multifunctional and mobile equipment.

Figure 7. Digital photofluorogram with a low-contrast shadow.

A CereTom 300 computer tomograph from NeuroLogica (USA) is intended for examining tissues of the head and neck. It may be employed in resuscitation and emergency departments, neurosurgical centers, X-ray surgical research centers, operating rooms, intensive care units, patient's hospital rooms, neurology units, newborn resuscitation rooms, maxillofacial surgery centers, ambulances, and dental offices.

Tekh-Astor (Russia) manufactures a highly stable X-ray Ekstravol't apparatus for industrial flaw detection. It is a cable type instrument with a ceramic-metal tube intended for industrial radiography and radioscopy at large X-raying depths (up to 102 mm of steel under standard conditions) in industrial and scientific laboratories, as well as in shopfloor conditions.

Balteau NDT (Belgium) developed a crawler — a self-contained self-moving X-ray complex intended for the quality control of welded joints of pipelines under construction (Fig. 8). The instrument moves inside a pipe, is located under the joint to be checked, and executes an exposure operation. An operator located outside of the pipe manipulates the crawler with an electromagnetic control system.

Light and mobile microfocus constant-potential microprocessor-controlled RAP-M instruments are intended for the nondestructive inspection of metal and nonmetal articles. Due to the presence of instruments with panoramic and side output of X-ray radiation, it is possible to check articles of complex configurations.

Small-sized pulsed MIB betatrons are employed for the radiographic quality control of materials and articles in nonstationary conditions: construction sites, ship slips, welded joints and stop valves for oil and gas pipelines, repair of power and boiler installations, bridge footings and other critical building structures, and casting and thick welded joints.

X-ray equipment is actively employed in security systems. For instance, a Kalan-2M stationary X-ray introscope is intended for the X-ray inspection of separate items (packages, small parcels, etc.) for detecting explosive assemblies, weapons and firearms, as well as other articles prohibited for carriage. The perfect radiation safety of the facility permits operating it as a means of entry inspection and accommodating it in working spaces (in enterprises and offices). Due to the use of a microfocal X-ray emitter and

Figure 8. BNDT crawler.

the presence of a translation object stage in the inspection chamber, it is possible to obtain geometrically magnified images of separate parts of the object under inspection.

A Norka portable X-ray TV unit is intended for the inspection of mail, luggage, furniture, and different everyday articles for the purpose of the detection of explosive assemblies and containers with dangerous enclosures. It provides a high throughput capability for a weak radiation effect on the milieu and service personnel.

Beyond that point, X-ray sources are employed in scientific research. For instance, an iMOXF attachment for scanning electron microscopes (SEMs) is a combination of an X-ray source and X-ray optics, which substantially broadens the SEM capabilities in the field of microanalysis. iMOXF permits performing X-ray fluorescence microanalysis, and dedicated software makes it possible to jointly process the data obtained in X-ray spectroscopic and X-ray fluorescence analyses.

Apart from laboratory modifications, there are also portable X-ray fluorescence analyzers intended for the solution of applied problems. For instance, X-MET — a portable X-ray fluorescent analyzer of the chemical composition of metals and alloys — is employed for monitoring steels and nonferrous metal alloys, sorting scrap metal and metals in storage areas, performing rapid analysis and determining grades of metals at every production stage, repairing and diagnosing equipment, performing rapid analysis of ores and soils, executing environment monitoring, and diagnosing and controling industrial safety.

5. Progress in X-ray equipment design

At present, the development of X-ray tubes is proceeding along two main lines: designing general-purpose devices and designing special-purpose tubes intended for the solution of particular problems. For instance, the scanning X-ray topography of crystals [25, 26] and the investigation of crystals using a widely diverging beam [27, 28] require X-ray tubes with a design radically different from that of general-purpose tubes [29].

The majority of X-ray tubes having found mass application have reached certain structural uniformity in the course of their development. Almost all of them consist of two electrodes — an anode and a cathode — soldered in a glass bulb and located on the tube axis opposite to each other. In most cases, the X-ray radiation emanates from the middle of the tube perpendicular to its axis.

However, there are several special-purpose tubes which structurally differ greatly from the 'normal' mass-application tubes. Among them are:

(1) tubes with an offset hollow anode, which are employed for abdominal therapy and X-raying hollow articles;

(2) tubes with a rotating anode, which permit obtaining a high short-time power for a small focus and are employed in certain kinds of X-ray diagnostics;

(3) soft X-ray tubes with a high output power, which may be applied in the investigation and employment of bactericidal and photochemical properties of X-rays;

(4) pulsed tubes used for the microsecond X-ray radiography of fast processes;

(5) miniature tubes for light transferable devices;

(6) high-voltage tubes for deep therapy and X-raying of critical thick industrial products;

(7) sharp-focus tubes — X-ray shadow microscopes.

The use of computer tomographs for investigating transverse brain sections was proposed in England in 1972 [30]. As is well known, obtained in ordinary X-ray radiography is a two-dimensional image, and the details existing in three dimensions overlap each other and distort the picture, which hinders obtaining the requisite information. In a computer tomograph, advantage is taken of an original method of obtaining images, which permits overcoming these difficulties: the object is exposed to transverse scanning by a collimated X-ray beam. The radiation behind the object is recorded by a detector array, and the measurement data are employed for synthesizing a half-tone image of the layer with the aid of a computer. The resultant image has no shadows and distortions attributable to other layers of the object under examination (Fig. 9). The information content of a tomogram is approximately 100 times greater than in the ordinary roentgenogram, all other conditions being equal [31, 32].

The next stage of the progress in computer tomography involved the development of computer microtomographs for the investigation of technical and some biological objects. They necessitated portable X-ray devices with X-ray tubes that were radically different in characteristics and design [33] from the X-ray tubes employed in medical computer tomographs [13, 30, 34]. To make them required microfocus X-ray tubes with a relatively high nominal voltage (100–150 kV), a high working beam intensity, and, as a rule, a tube-end beam output.

Similar requirements also had to be met by X-ray tubes intended for the microdefectoscopy of small articles, including those possessing cavities. In the latter case, the tube was to have an offset anode with a long flight tube.

Therefore, a demand for X-ray tubes for technical computer microtomographs, special-purpose X-ray diffraction analysis, microdefectoscopy, dental examinations, etc. has arisen.

Among the important practical problems arising in the use of X-ray radiation is the search for a method for controling beams. The energy and direction of propagation of charged particle beams may be easily changed under the action of electric and magnetic fields. Light beams may be manipulated by using Kerr and Faraday cells, as well as flexible optical fibers. At the same time, the possibilities of controling X-ray beams are severely limited. There are presently known two fundamental phenomena which enable affecting the propagation of X-rays: the Bragg reflection, and the total external reflection of the radiation [35]. An important supplement to them is the Borrmann effect [36].

Proceeding from these phenomena, an arsenal of means for X-ray beam transformation was created: Bragg–Fresnel zone plates [37], X-ray polycapillary devices [38], crystal [39] and multilayer [38] monochromators, and X-ray refractive lenses [41]. Quite recently, X-ray waveguide-resonators have appeared [42]. All these devices are characterized by the presence of direct external action on X-ray beams. Owing to the smallness of X-ray wavelengths, however, this action turns out to be insignificant. And so, the idea of using indirect methods of X-ray beam modification, for instance, by action on the interference field of the standing X-ray wave induced by these beams, seems highly attractive. E Egorov and V Egorov [43] suggested that this approach to X-ray beam modification be realized using a waveguide-resonator [44].

Examinations in stomatology and maxillofacial surgery necessitate specialized microfocus X-ray tubes which enable obtaining panoramic images.

X-ray tubes [45, 46] operating in a static regime are known to be used in medicine in the intracavitary irradiation of tumor tissues and in engineering for the X-ray imaging of complex mechanisms and devices for the internal accommodation of the radiation source. A drawback of these tubes is the existence of a hot cathode in the structure and the need for a cooling system. Such tubes quite often contain structural elements for electron beam focusing, which increases the tube dimensions.

It is possible to miniaturize the X-ray source and extend the area of its application by employing pulsed X-ray tubes with a cold cathode. An operation of the sealed-off pulsed X-ray tube has been reported [47], which comprises an evacuated glass holder, a multispike field-emission cathode, and a cone-shaped anode made of a heavy metal and located on the tube axis opposite the cathode. The tube operates for a voltage pulse amplitude higher than 100 kV and a pulse duration of about 10 ns. Among the disadvantages that limit the application of this tube are its relatively large dimensions (25-mm diameter, and 140-mm length), the complexity of assembling the cathode, which consists of a large number of thin spikes, and a short service life.

In 2000, the All-Russian Research Institute of Experimental Physics (Sarov) obtained a patent for a miniature pulsed X-ray tube, which produced short soft X-ray pulses (with a duration of about 1.5×10^{-10} s) with photon energies in the range between 30 and 90 keV [48]. The technical result was achieved due to the fact that the target was separated from the window in the pulsed X-ray tube, which comprised a metal case with a shoot-through target and a window for X-ray radiation output, a cathode, and an interior insulation element. The target was secured in the tube internal chamber using two cylindrical rings with a tooth and a groove, so that the ratio between the target–cathode gap and the external diameter of the cathode ranged from 1:20 to 1:5. The insulation element can be made in the form of a ring.

In recent years, the radiation of soft X-ray and extreme ultraviolet (UV) ranges has found wide application in reflectometry, spectroscopy, and nanolithography. Laser

Figure 9. Comparison of the information content of a roentgenogram (a) and a tomogram (b) of a knee joint.

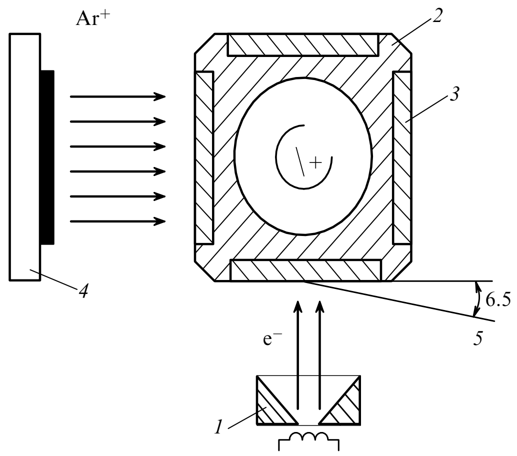

Figure 10. Schematic of an X-ray tube with an interchangeable cathode: *1* — electron gun, *2* — water-cooled holder, *3* — soldered target, *4* — ion source, and *5* — X-ray beam.

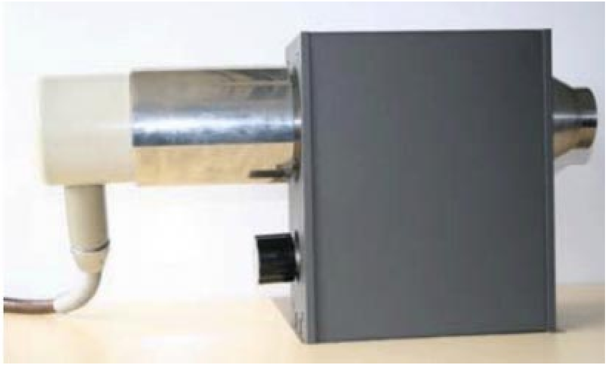

Figure 11. Dismountable X-ray tube.

plasmas and gas discharges are the more frequently used laboratory sources of extreme UV radiation. However, they suffer from several drawbacks significant for precision reflectometric measurements. Among them are the contamination of optics (by the products of erosion of the target in a laser-plasma source and the structural elements in gas-discharge sources), the complex spectral composition of radiation (the presence of close intensive lines gives rise to an appreciable background, which substantially affects the measurement accuracy), and the difficulty of source tuning to a different spectral range (moving from 100 to 1000–2000 eV will involve a significant change in power source requirements) [49].

X-ray tubes are devoid of these disadvantages. However, tubes operating in the soft X-ray range have several special design features, among which is the necessity of operating with a few spectral lines (which requires regular changes of the anode material) in the absence of frequent contacts of the cathode and anode units with the atmosphere in a number of devices. To alleviate these drawbacks, an X-ray tube design (Fig. 10) was proposed at the Institute for Physics of Microstructures, RAS, in which thoriated tungsten is used as the hot cathode material and there is an ion source intended for target cleaning by the ion-beam etching technique. In the course of operation, the target is changed by turning the target holder through 90° without access of atmospheric air. According to thermal calculations [51], the highest power of the X-ray tube is liable to reach 500 W.

An X-ray tube with several excitation sources (Cr, W, and Rh) was developed in Japan in 2008 [52]. By displacing the anode, it was possible to select the excitation source with retention of the vacuum. The focal spot size of this X-ray tube was equal to 10 μm at a voltage of 50 kV and a current of 0.5 mA. The convenience of applying this X-ray tube for X-ray fluorescence analysis (the excitation of different elements, depending on the target material) was confirmed with three standard samples of powder materials: TiO_2, Co, and Zr.

At present, several companies produce nanofocus X-ray sources, which are employed in different defectoscopic facilities having focal spots 0.5–1 μm in diameter on tungsten hot cathodes at 20 kV. Tubes with such focal spots permit resolving low-contrast features measuring several hundred nanometers and produce magnified images.

To obtain focal spots 10–100 nm in diameter, the accelerating voltage should be lowered to 3–5 kV to shorten the effective electron range in the target, which is the reason why the size of the focal spot (of the X-ray source) exceeds the electron beam dimension. This will entail a significant lowering of the power and power density of the X-ray source. In the image formation of nano-dimensional objects, this lowering is partly compensated for by the higher absorption of the soft X-ray radiation by nano-dimensional object's features.

A nanofocus X-ray source with a dismountable X-ray tube for accelerating voltages of 3–40 kV was developed as a solution to this problem. A system of three magnetic lenses and an electron gun at a tungsten cathode, optimized for low accelerating voltages was employed to focus the electron beam. The minimal size of the electron beam that the system was able to form measured 10 nm. The lens system was accommodated in the same framework (170 $\times$ 350 $\times$ 300 mm, and weight $\sim$ 30 kg) with a magnetic-discharge pump and manually controlled seals (Fig. 11).

In the test of the first version of a pilot sample of the transmission X-ray microscope with a dismountable tube, a resolution of 0.1–0.2 μm was inferred from the scan pattern of a test target in the transmitted X-ray radiation at 10 kV [53]. In the projection regime, a resolution of 0.3 μm was achieved with an X-ray detector at 25 kV.

Due to recent achievements in X-ray optics, it has become possible to reduce by at least two orders of magnitude the intensity of X-ray sources employed in medicine and industry, while obtaining the same results. This creates a need for new-generation small-sized low-power microfocus X-ray tubes. By replacing a hot cathode with a field-emission cathode, it is just possible to decrease the sizes of the X-ray source and its power consumption [54]. That is why recent research has been focused on carbon fibers and carbon nanotubes as likely electron sources in miniature X-ray tubes.

6. Use of carbon materials as cathodes

A classification of the main modern techniques of making field-emission cathodes from two large classes of carbon materials (Fig. 12) was discussed in Ref. [54]. The first is a material of industrial production, while the second comprises directly formed field-emission structures. The paper also outlines the special features of concrete methods of making field-emission cathodes.

Knapp et al. [55] considered the influence of electron source parameters on X-ray tube characteristics in relation to

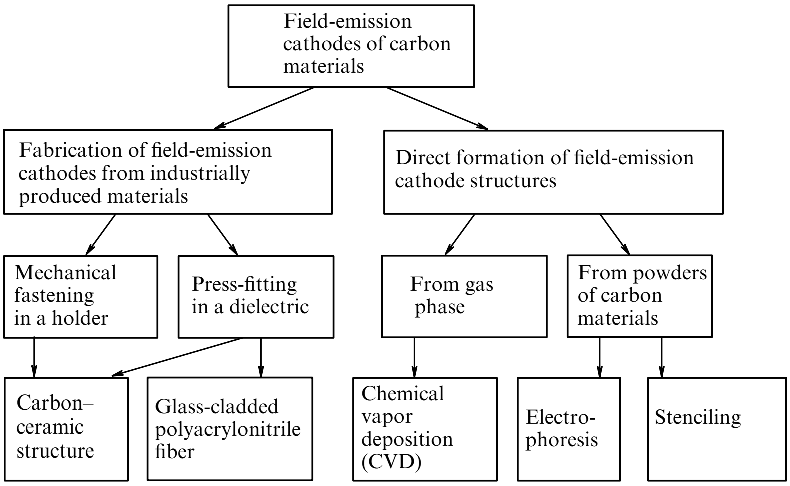

Figure 12. Classification of the main fabrication methods for field-emission cathodes of carbon materials.

different types of field emission cathodes; a comparison was also made with hot cathodes.

The authors of Ref. [55] propose the employment of one or several pointed field-emission cathodes. However, it should be taken into account that the emergence of a spark discharge inside the X-ray tube will entail an irreversible disruption of the emitter spike. That is why it was decided to employ carbon fibers [56] and carbon nanotubes [57] for likely materials of field-emission cathodes, as the most stable and capable of self-recovery.

The staff members of the Moscow Institute of Physics and Technology (MIPT) proposed a fabrication method for high-efficient field-emission cathodes of carbon–nitrogen nanofibers [58]. The threshold electric field strength for these field-emission cathodes is substantially lower than for field-emission cathodes made of multilayer carbon nanotubes and known carbon–nitrogen nanomaterials. The method involves the growth of nitrogen-bearing carbon nanofibers by resistive heating of graphite in an atmosphere of nitrogen or a high-pressure nitrogen–argon mixture and their deposition on a specially prepared graphite substrate in the working volume.

The developed method of fabricating field-emission cathodes permits synthesizing carbon–nitrogen nanofibers in a high-pressure apparatus directly on the working substrate, without invoking intermediate operations. This circumstance simplifies the technological cycle and lowers the fabrication cost of the field-emission cathode. In this case, a preliminary substrate preparation provides enhanced adhesion of the carbon–nitrogen material and its service durability in the course of field-emission cathode operation. As a result, applying the method developed instead of the standard stenciling technology permits attaining higher emission current densities, as well as improving the operation stability and the service life of the field-emission cathode.

The results of investigations into the field-emission properties of cathodes made of graphite foil were outlined in paper [59]. The emission centers on its surface were formed using a laser marker. The experiments performed in work [59] demonstrated the possibility of making field-emission cathodes with a uniform surface structure. Topographically different field-emission cathodes were manufactured: craters ~ 60 μm in diameter with center-to-center distances equal to the diameter, and a mesh with a cell width of 50 μm.

Investigations of the emission properties showed that the best characteristics were exhibited by those cathodes with a topography in the form of craters. Cathode stability investigations revealed that the emission parameters of the cathodes with surface topography in the form of craters are stabilized in the course of training involving an increase in current load. After this training, the field-emission cathodes under investigation did not undergo degradation, for a cathode take-off current density of about 1 mA cm^{-2}.

Carbon nanotubes (CNTs) were discovered about 20 years ago [60]. Due to the special features of their chemical bonds and an ideal geometry, they exhibit many unique properties [61], among which are the unassisted maintenance of rather sharp tips and a high aspect ratio. This is the reason why CNTs possess a substantial field enhancement factor, with the consequence that lower threshold intensities are required to yield field emission, when they are employed [62]. The energy spread in such cathodes amounts to 5 eV, and the angular spread of emitted electrons is less than 5° relative the field direction.

Lamanov et al. [63] came up with the idea of growing nanotubes by low-temperature ethanol vapor deposition. Gas-phase deposition refers to one of the most efficient fabrication methods for flat field-emission cathodes, and it permits obtaining different carbon structures on a cathode substrate. The resultant carbon coatings may be a diamond-like films [64], amorphous graphite [65], or various carbon structures including CNTs [66], depending on the deposition conditions. Experiments have demonstrated the possibility of synthesizing CNTs by low-temperature (the substrate temperature is equal to 500 °C) ethanol vapor deposition. Stability investigations of the resultant cathode samples showed that the field-emission properties of the field-emission cathodes under investigation underwent only insignificant changes for a take-off current density not exceeding 80 μA cm^{-2}.

One of the main characteristics of field-emission cathodes is their service life. Chesov et al. [67] studied the degradation processes of field-emission cathodes fabricated from carbon nanotubes and proposed a method for analyzing the time dependence of the current and voltage, as well as criteria for comparing field-emission cathodes with substantially different working voltages. As shown in paper [67], the fabrication method of a CNT field-emission cathode (stenciling or electrophoresis) has no effect on its service life, and the degradation rate depends primarily on the cathode current. The emission properties of field-emission cathodes were determined to vary in a deenergized state. This is due to the adsorption and desorption of residual gas molecules in the device, which lower the electron work function and are responsible for the emergence of surface portions with a high degradation rate immediately after switching it on. For long field-emission cathode operation (over 100 h), the main cause of degradation relates to a decrease in the emitting surface area, while the form factor is hardly changed.

Carbon nanotubes may serve as high-current sources. From a single one-layer CNT, it have been possible to obtain a stable emission current of over 1 μA [68]; currents with a density of over 1 A cm^{-2} have been obtained from macroscopic CNT cathodes [69]. Owing to these properties, carbon nanotubes hold promise for many technical problems. The possibility has been demonstrated of using them as field-emission cathodes in flat field-emission displays (FEDs) [70], light sources [71], and discharge tubes intended for overvoltage protection [72].

However, it is well known that disruption or self-healing of the working cathode surface occurs under its bombardment by ions of the residual gas in field-emission devices [73]. In cathodes made of graphite materials, self-healing of the working surface occurs under the ion bombardment due to the formation of a statistically equilibrium microstructure of emission centers. That is why ion bombardment for graphite-based cathodes refers to the dominating mechanism of both the formation and disruption of the working surface.

As shown in Ref. [74], the enhanced stability of graphite cathodes is due to a large number of existing emission centers. In the making of emission centers, however, attempts to obtain microstructures with the same geometrical dimensions seldom meet with success [75]. For this reason, the emission of electrons in the first switching proceeds non-uniformly over the cathode surface. However, the number of operative emission centers increases after long cathode operation at a current density of 0.5 mA cm^{-2} for 10–40 h, because the formation of a stable working surface relief and statistical equalizing of the geometrical parameters of emission centers occur due to disruption of those centers with a high field enhancement factor [75, 76]. Furthermore, the weakly bound components are removed in the course of operation and those surface microrelief configurations that are most dynamically stable stands out. In this case, all fragments are removed from the surface due both to ion bombardment and under the action of a ponderomotive load. For instance, an investigation was carried out of the emission properties of carbon field-emission cathodes made of carbon fiber bundles and high-strength MPG-6/MPG-8 graphites [75]. It was shown that the current take-off changed significantly during the first 10–50 h of operation; subsequently, the emission current remained practically stable. SEM surface investigations of field-emission cathodes with different operation durations proved that the emitting surface underwent restructuring under ion bombardment in the course of cathode operation. This transition process is referred to as cathode preparation or cathode training. Therefore, if cathodes made of graphite materials are to meet the requirements on emission current stability, efficiency, and emission center distribution uniformity, a special technique is required for their training. In this case, the temporal and technological expenditures for cathode training have to be minimized for substantially reducing their cost in mass production.

Sherstnev et al. [77] came up with the idea of simulating the regime of current cathode training [75] by way of plasma etching of the cathode surface and then putting the cathode to a service-life test. It was shown that by subjecting the cathode surface to an intensive ion etching for a very short time, it was possible to simulate the changes of the working surface of a cathode that had been functioning for a long time. Experiments have given evidence that the number of emission centers in the first switching after the cathode ion etching increased by approximately 3.5 times in comparison with an unetched cathode. The SEM surface image of a cathode which has been working for 720 h is visually similar to the cathode surface image after the etching which simulates the 720-h operating period.

7. X-ray tubes with field-emission cathodes

In 2003, a patent was registered in the USA for an X-ray source comprising the field-emission cathode [78]. The cathode of this X-ray source had to be formed at least partly of nanostructured materials, affording the emission current density amounting to 4 A cm^{-2}. Therefore, it is possible to obtain a substantially higher emission current density than with hot cathodes. The high energy efficiency of such a source is realized due to a sharp lowering of anode heating.

In 2003, MIPT researchers applied for a patent on a miniature X-ray tube with a field-emission cathode made of a bunch of carbon polyacrylonitrile fibers [79]. This tube was made according to a triode scheme and comprised, apart from the cathode and the anode, a control electrode (Fig. 13). The high efficiency of its design solution was borne out by the tests conducted. A tube at a voltage of 50 kV and a current of 100 μA worked for more than 10,000 h without failure, which testifies to its high reliability. In this case, the instability of tube characteristics fell within 1%.

This invention affects on X-ray tubes with a field-emission cathode made on the base of carbon materials; it may be employed as an X-ray source in flaw detectors, examination instruments, medical X-ray devices, and diagnostic X-ray spectroscopic facilities.

The X-ray tube (see Fig. 13) comprises a vacuum envelope (*1*), a field-emission electron gun (*2*) accommodated in it together with an anode (*3*), and an X-ray output window (*4*). As for the field-emission electron gun, it contains a field-emission cathode (*5*), a cap (*6*) with an electric outlet (*7*), a dielectric bead (*8*), and a contact unit (*9*) of the field-emission cathode.

A carbon fiber bunch (*10*) is enclosed in a shell (*11*) for providing the orientation of the carbon fibers, their mechanical fastening, and vibration resistance. The shell is made of a conductive or semiconductive material, for instance, of a conductor, a semiconductive glass, or a surface-metallized dielectric, which rules out an accidental change in the shell potential relative to the carbon fiber bunch during X-ray tube operation. The carbon fiber bunch projects outward the shell on the emitter side. There is a diaphragm in the form of an opening (*12*) in the end wall of the cap which serves as a tube control electrode.

The contact unit (*13*) of the field-emission cathode is made of an electroconductive material, for instance, of an electroconductive paste (based on a powder of Ag, Al, or other conductors) or aquadag (a suspension of finely dispersed

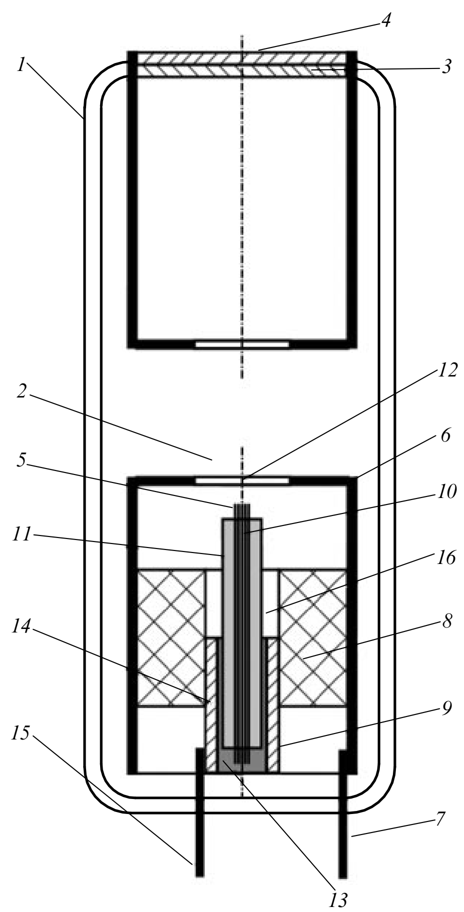

Figure 13. Design of an X-ray tube with a field-emission cathode of a carbon fiber bunch.

graphite) deposited onto the end of the carbon fiber bunch opposite the emitter and onto a part of the shell squeezed by a metallic ferrule (*14*) which can be made in the form of a whole cylinder or a cylinder cut along a plane passing through its axis. The ferrule (*14*) has an electric (cathodic) outlet (*15*) from the evacuated envelope and is rigidly connected to the dielectric bead using, for instance, a glass cement, glue, or a solidified resin in such a way that the central axes of the ferrule, the carbon fiber bunch enclosed in the shell, and the opening of the dielectric bead are coaxial with the electron-optical system of the X-ray tube. The tube is assembled in such a way that a cavity (*16*) is formed between the inner walls of the opening in the bead, the shell, and the upper end of the contact unit. This cavity prevents the emergence of surface electric conduction due to the formation of dispersed and continuous semiconductive and conductive films and tracks on the surfaces of the cathode shell, the dielectric bead, and the control electrode in the thermal vacuum processing of the X-ray tube and the sputtering of the cathode material subjected to ion bombardment. Furthermore, it is necessary to rule out the breakdowns between the X-ray tube electrodes, which emerge in the training and operation of the X-ray tube.

The X-ray tube operates on applying to the anode a high positive potential relative to the cathode and a voltage between the cathode and the control electrode, which gives rise to field electron emission from the ends of carbon fibers protruding outward the shell. Numerous fibrils — 250–1000-Å long tetragonal carbon crystal objects 20–50 Å in diameter — which are extended along the fiber axis rise to the surface of the ends of the carbon fibers. They are centers of the field emission from the carbon fibers. The electrons field-emitted from the cathode emanate through the opening in the end wall of the cap which fulfils the function of a control electrode, and find themselves in the strong accelerating electric field between the anode and the cap. The diaphragm opening in the cap allows field-emitted electrons to fly only towards the anode, preventing them from flying from the cathode to the dielectric part of the X-ray tube envelope. As a result, the electric strength of the tube is enhanced, because the probability of charging and breakdown of the dielectric tube envelope is ruled out.

The accelerated high-energy electrons hit the anode to give rise to X-ray emission.

Varying the control electrode potential changes the electric field strength at the end surface of the emitting carbon fibers and thereby adjusts the X-ray tube current and the intensity of X-ray radiation for an invariable cathode–anode potential difference.

More recently, other electron gun designs have been proposed at MIPT for different vacuum devices, which possess better characteristics as regards controllability, assembly simplicity, and other parameters [80–82]. To achieve a more stable operation of the field-emission cathode, Eroshkin et al. [83, 84] proposed applying the method of plasmachemical emitter etching in a corona discharge.

Miniature X-ray sources hold promise for application in therapy, because the irradiation can be turned on at the requisite point in time (when the source is pointing at the object under examination) and turned off on accumulation of the desired dose, unlike those X-ray sources which emit continuously and may constitute a threat to patients and the medical staff operating the devices. To this end, in the treatment of, e.g., surface arteries of the heart, it is required to have a catheter no more than 1 mm in diameter. For other purposes, like the treatment of cancer of the esophagus or rectal cancer, diameters of up to 5 mm are acceptable. Several designs which employ X-ray tubes with field-emission cathodes as electron sources are proposed in the literature for these purposes [85–87]. For a cathode material use is made, for instance, of nanodiamond-carbon [85] or carbon nanotubes [86, 87].

Busta et al. [88] proposed prototypes of such tubes ranging from 1 to 5 mm in diameter, whose housing is made of quartz or borosilicate glass. Since these miniature X-ray tubes operate at voltages of 15–40 kV, it is required that no breakdowns, including surface ones, occur during service. That is why, apart from the use of field-emission cathodes, special emphasis in the development of these tubes was placed on their insulation and placement in catheters.

A new design of a subminiature X-ray tube with a carbon nanofiber cathode was developed at the Nagoya Technology Institute (Japan) in 2004 [89]. The assembly consists of a 5-mm long glass tube 2 mm in diameter, with a stainless steel tube with an internal diameter of 0.5 mm welded into it. A cathode of molybdenum wire 0.5 mm in diameter is inserted into the steel tube, and a layer of vertically oriented nanofibers is grown by chemical deposition onto the wire (Fig. 14). As a target, use was made of a $\sim$ 3-μm thick copper film deposited onto a 0.1-mm thick aluminum platelet. The electric potential applied to the cathode was varied in the 10–15 kV range, which provided an electron emission current of 50 μA. For higher currents, the target was damaged. The measured emission current–voltage characteristics of the

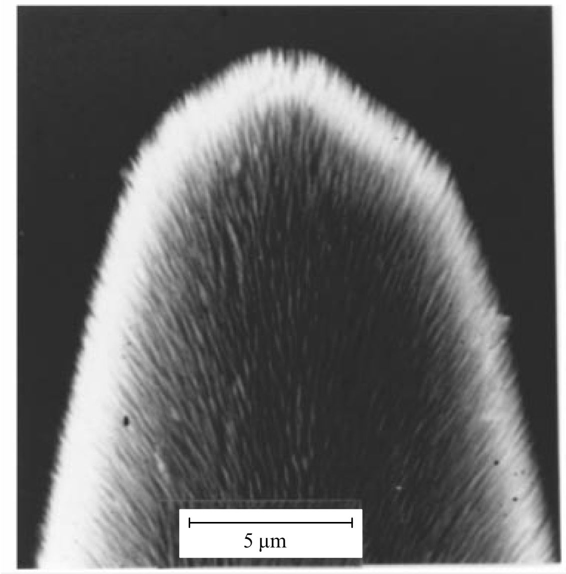

Figure 14. Molybdenum wire cathode with carbon nanofibers grown on it.

emitter agree nicely with the classical Fowler–Nordheim dependence, which supports the field electron emission mechanism. The X-ray emission spectrum consisted of two lines (K_α and K_β) with peaks near 8 and 9 keV, respectively, superposed on the continuum arising from the electron bremsstrahlung. When testing the X-ray tube, it was possible to obtain high-quality images of biological objects (tree leaves, in particular) with a high resolution unattainable when utilizing conventional commercial X-ray units.

A miniature X-ray tube of a triode design using a CNT-based field-emission cathode was presented in Ref. [90]. The authors managed to achieve a control voltage of less than 1000 V for a focal spot size of 1 mm, which makes this tube suitable for X-ray fluorescence analysis.

Recently, researchers at the Korea Institute of Science and Technologies developed an X-ray source with a resolution higher than 5 μm [91]. This result was achieved by the use of a special cathode configuration, in which the electron-emitting nanotubes are deposited on the surface of a sharp tungsten tip with a radius of curvature of ~ 5 μm. The X-ray tube design is schematically shown in Fig. 15.

The tip of a 250-μm thick tungsten wire with a pointed apex was made by electrochemical etching. The carbon

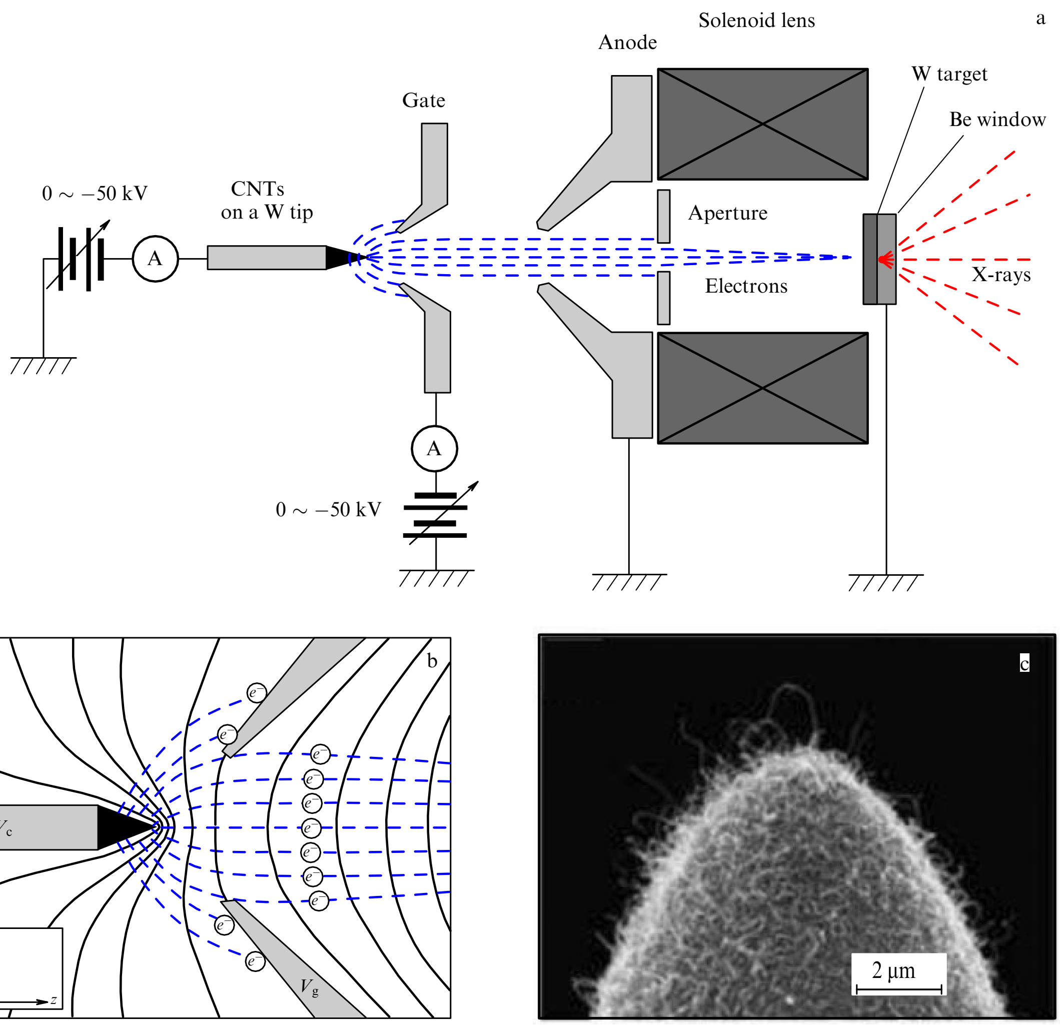

Figure 15. (a) Schematic of an X-ray source comprising an electron gun, a lens in the form of a solenoid, and a shoot-through target; (b) calculated electron trajectory, and (c) microimage of the cathode whose surface is uniformly coated with multilayer CNTs about 50 nm in diameter and approximately 1 μm in length.

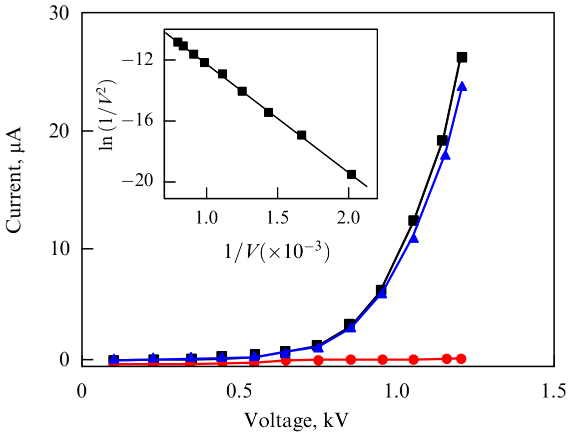

Figure 16. (Color online.) Emission characteristics of a CNT-based cathode and transport characteristics of an electron gun: (black) squares — emission current, (red) dots — current lost at the control electrode, (blue) triangles — current reaching the target. The inset shows the current–voltage characteristic in Fowler–Nordheim coordinates.

nanotubes on the surface of the tip were grown by plasma enhanced chemical vapor deposition (PECVD) with nickel as a catalyst. To improve adhesion of the nanotubes, a buffer layer of titanium nitride was deposited onto the etched surface of the tip. The degree of uniformity of the nanotube coating of the tip is demonstrated in Fig. 15c.

To weaken the spherical aberration effect, use was made of an electromagnetic lens in the form of a solenoid, as well as an aperture stop from 4 to 10 mm in diameter, which was placed prior to the beam entry to the lens. To ensure that the electron collisions with the atoms of the target material do not increase the X-ray beam divergence, advantage was taken of a shoot-through type target whose thickness was much smaller than the characteristic electron range in the material. The target comprised a 500-nm thick beryllium film 20 mm in diameter with a sputtered tungsten layer. The measurement data on the emission characteristics of the CNT-based cathode and the transport characteristics of the electron gun are given in Fig. 16.

The measurements were carried out at a cathode voltage of -40 kV and a varied voltage of the grid spaced 0.25 mm from the cathode. The electric field strength was equal to 1.6 V μm^{-1}, which provided an emission current density of 10 mA cm^{-2}. The electric field enhancement factor obtained from the processing of the Fowler–Nordheim law was equal to 2700. So strong an enhancement is achieved by employing a special cathode configuration, whereby CNT emitters are located on the pointed tungsten tip rather than on a flat surface. The emitting surface area is estimated at 1.6×10^{-6} cm^2, which approximately corresponds to the area of a hemispherical tip with a radius of curvature reaching 5 μm.

The characterization tests of the X-ray tube described above were made using a test grating which contained 6-μm wide gold strips deposited onto a substrate and spaced at 25-μm intervals. The test data allow the conclusion that this instrument has a resolution below 5 μm.

8. Conclusion

At present, microfocus X-ray tubes and portable equipment based on them are efficient means for investigating the structure of different objects at the microlevel. They are employed for microdefect detection, microtomography, crystallographic research, quality control of industrial products, medical examinations, and many other purposes. In the development of microfocus X-ray tubes one can notice a trend towards making purpose-oriented devices which best meet the requirements imposed by specific investigations.

The central type of modern microfocus X-ray tubes are devices with a transmission anode devoid of special coolers. The main trends in the development of devices with a transmission anode involve a further increase in voltage and power for a decrease in focal spot size, an increase in tube efficiency, and a decrease in its dimensions. This may be achieved by replacing hot cathodes with carbon nanomaterial-based field-emission cathodes.

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