GENERAL CONSIDERATIONS FOR DESIGN AND CONSTRUCTION OF TRANSMISSION ELECTRON MICROSCOPY LABORATORIES

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ABSTRACT

One of the main points in facilities for nanoscience research work, besides the clean areas, and other special zones, is that which refers to the precision laboratories for the location of optical instruments and other research and analysis equipment, as Transmision Electron Microscopes (TEM), which require specific environmental and technical conditions for their correct operation. This paper reviews the general considerations and the limit values associated with acoustic vibration, electromagnetic interference and thermal fluctuation phenomena that affect the performance of a TEM. The recommendations that are reviewed may be also generally useful to design rooms where scanning electron microscopes (SEM) and different types of precision instruments can be located.

Keywords: Transmission Electron Microscopy, Design, Nanoscopy, Laboratories, Environmental Conditions.

CONSIDERACIONES GENERALES PARA EL DISEÑO Y CONSTRUCCIÓN DE LABORATORIOS PARA MICROSCOPIA DE TRANSMISIÓN ELECTRÓNICA

RESUMEN

Uno de los principales aspectos en instalaciones para el trabajo de investigación en nanociencias, además de las zonas limpias, y otras áreas especiales, es el referido a la ubicación de instrumentos ópticos y equipos de análisis e investigación, tales como microscopios de transmisión electrónica (*Transmision Electron Microscopes*: TEM), los cuales requieren de condiciones ambientales y técnicas específicas para su correcta operación. Este artículo revisa las consideraciones generales y los valores límite asociados con fenómenos de vibración acústica, interferencia electromagética y fluctuaciones térmicas, que afectan el desempeño de un TEM. Las recomendaciones que son revisadas pueden ser además de utilidad general para el diseño de cuartos que albergan microscopios de barrido (*Scanning Electron Microscopy*: SEM), así como diferentes tipos de instrumentos de precisión.

Palabras claves: Microscopía de Transmisión Electrónica, Diseño, Nanoscopía, Laboratorios, Condiciones Ambientales.

INTRODUCTION

According to researchandmarkets.com in 2011 the world market for electron microscopes was forecast to exceed US$4.3 billion in annual sales by 2017, due to increased demand for sophisticated, high-resolution and precision imagery across a range of existing and new markets [1]. Areas of demand for Transmission Electron Microscopies (TEMs) include semiconductors, liquid crystals, chemistry (silicone and polymers), data storage, memories, medicine and bioscience, among others. The market has expanded with the "nanotechnology boom" when it was marketed as a device for basic research purposes to public research institutions. The ratio of demand from universities and public research institutions and that from private sector is estimated at a 6:4 split, as TEMs are most often used for basic research purposes. In this regard, one of the main points in facilities for nanoscience research work, besides the clean areas, and other special zones, is that which refers to the precision laboratories for the location of optical instruments and other research and analysis equipment, as transmission electron microscopes, which require specific environmental and technical conditions for their correct operation.

Although the technical treatment for precision laboratories has gained greater importance as the number of nanotechnology research centers has increased worldwide in the last 10 years, the associated technical literature is still scarce and relatively scattered. As example, while the United States National Nanotechnology Initiative (NNI) began in 2002, during the second half of 2012 the United States Institute of Environmental Sciences and Technology (IEST) reported that the document IEST-RP-CC200: *Planning of Nanoscale Science and Technology Facilities: Considerations for Design, Construction, and Operation* was in preparation. This is why in most cases the consolidation of knowledge related to the design, construction and operation of research facilities at the nanoscale level are based on the collection of specific experiences and reports by researchers from universities and research centers throughout the world, who are responsible for the installation of precision equipment.

Besides the SPIE proceedings on Buildings for Nanoscale Research and Beyond, and other documents from some architectural and design firms available in the Internet, there are only some specialized articles, especially in microscopy and metrology journals, related to the environmental conditions and requirements to design the rooms to house transmission electron microscopes. Indeed, the proliferation of microscopes with greater capabilities, including image filters and specialized hardware to correct spherical aberration (allowing greater spatial and energy resolution) has promoted the need to build laboratories and suitable spaces for practical work and research. In other words, to the extent that resolution is improved, the performance of precision microscopes is strongly influenced by environmental factors. Such environmental factors are reviewed in this article, including magnetic fields, vibrations, changes in barometric pressure, variations in the cooling – water temperature and grounding systems, among others [2]. This leads to the preliminary conclusion that facilities are truly a part of the microscope and must be budgeted together.

Environmental Issues. As a first factor, the vibrations like sound waves can reach the microscope through the air (acoustic vibration), across the ground, from outside traffic, near industrial processes, natural micro earthquakes, as a result of the operation of the auxiliary equipment required for the microscope, and even by simple factors such as the movement of the operator's seat. Vertical vibration of the specimen in the objective lens helps to disperse the focus and may limit the resolution. Additionally, the horizontal vibrations generate image defects and also limit the resolution in more than one direction. This is why high resolution and controlled focus can be achieved only under controlled conditions. But, as an important reference, precision microscopes are much more sensitive to low – frequency vibrations (LFV), which are much more complex to eliminate from the equipment's work environment. The conventional, as well as the atomic resolution spectroscopy (STEM, EELS, FEI), and images obtained by HAADF and LAADF modes, can be affected by uncontrolled vibrations during the time required for data acquisition (~10s or more). Essentially, the serial nature of the image – acquisition process, together with possible oscillations from the environment, may cause images to appear distorted in the case of the STEM mode, while in the conventional recording mode used in TEM, instabilities cause the loss of contrast and resolution [3]. Besides, even if the present generation of microscope columns, as well as capacitors for lighting systems, are designed to absorb certain oscillations and to protect the thermoionic source, sometimes small oscillations can impact the performance of the emission gun when working at elevated resolutions (~< 0.2nm). This is critical to the formation of small electron probes in the STEM mode [4], as well as for the coupling mechanism between the objective and the imaging lens in conventional TEM operations.

Second, electromagnetic interference (EMI), as well as stray magnetic fields, both from DC and AC sources, may cause aberration during the PCI (HRTEM) mode, distortions in the case of the STEM and the loss of energy resolution in the EELS mode. This type of environmental instabilities also constitutes limiting factors to obtain distortion – free images in the ADF mode. Similarly, spectroscopic systems, implemented as complementary accessories to the original instrument column, are particularly sensitive to changes in the magnetic field, which can penetrate through cameras and recording systems or unshielded display (~1eV/mG [5]). In general, any material with a high magnetic permeability, which is placed near the spectroscope, may divert the field lines and becoming a source of interference. Even metal wheels of a typical office chair might cause similar effects as those caused by AC fields.

As a general mitigation strategy, precision microscopy rooms are often located in basements, although sometimes right in the path of all grounding systems. These factors, as well as others that are presented in this paper, require that building designs for precision microscopy and the definition of different mitigation mechanisms, be developed in detail and by specialized firms.

As the third aspect, the fluctuations in air temperature may also cause small drifts or shifts, both in the specimen and the electron microscope, as well as in the positional tolerances for various components, including lenses and other elements. Then, an inadequate design of the cooling systems for the microscope room could affect the performance of the instrument. For instance little shifts in the filter opening. In particular, image reconstruction from focal series and from elementary mapping requires multiple exposures in the same study area of the specimen. For this reason, the sample should not experience any movement for several minutes, which must be ensured by a strict control of temperature and the possible LFVs in the precision room where the microscope is located.

To complement the general environmental issues, the barometric pressure of the room may be influenced by the opening and closing of the doors, generating effects in the sample – holder and possible shifts (up to 1 Å/Pa [3]). Also, in the laboratories where the specimens must be transferred to cooling stages and/or maintained at the temperature of liquid nitrogen, it is necessary to control the room humidity, which should be kept adequately low so as to prevent contamination by frost or the presence of ice (~< 15% of relative humidity).

As a preliminary conclusion it is key and also strategic – from an economic point of view – to pay attention to the preliminary design of rooms for precision microscopy, on the other hand a progressive process of mitigation of environmental conditions could be developed, in order to achieve an adequate level of operation for an electron microscope [3]. It is worth noting that these processes are unusual if the building is adequately design in advance and sufficient mechanisms for environmental mitigation are implemented from the beginning, to guarantee all performance levels that the instrument manufacturer ensures.

Design Tools and Mitigation Mechanisms. Most laboratories intended for the location of precision instruments for nanoscale research keep in mind similar design approximations to minimize the adverse environmental conditions previously outlined. Listed below are various alternatives:

Acoustic Noise: Factors, such as noise, acoustic vibrations and the heat generated by auxiliary equipment, are usually minimized by separation (through walls). Also, the noise and heat generated by computers can be minimized using solid – state amplifiers, which allow extending keyboard cables, mouse and screens as far as possible from the instrument(s). In general, it is necessary that the walls of the microscope room and that of other precision instruments, as well as the walls of the room where the auxiliary equipment is located, are designed and built to ensure "zero–acoustics", paying special attention to shielding for low frequencies, which – being the most complex to eliminate – are the greatest condition that affect the microscope column. As an important fact, the materials commonly used to reduce the noise, such as polyurethane or other kinds of foam, are virtually ineffective for bass in the 0–125 Hz band. To get an idea of the factor of which we are speaking, a sound wave to no more than the second octave (<32Hz), will have a wavelength of close to 10m, similar to the dimensions used in the main room of the instrument.

In practice, it is common to use a plastic layer initially that wraps around the perimeter of the precision room(s), including walls and ceiling. Later, it is recommended that drywall be installed in each wall of the room(s). Similarly, both the top of the wall, as well as its base, should be sealed to prevent the transmission of sound and air. A third layer may consist of an insulating and sound – absorbing material, usually fiberglass, placed between the walls and ceilings. Depending on the location of the room(s), the thickness of the fiberglass must be selected in order to reduce noise transmission; which usually has a good performance in the case of the attenuation of sounds with frequencies below 100 Hz. Finally, a topcoat layer usually employs a layer or board of sound – absorbing vinyl.

The previous options are globally useful for the precision room. However, to absorb low – frequency sound, the recommended experience consists of also leaving an empty space between the material or main layer of attenuation and the wall. A thickness of 15 cm has shown remarkable effects [5].

At the policy level, for any mitigation option that would ensure zero – acoustics, the ASTM E90 standard, which covers the classification of sound transmission through the air, is recommended. The classification consists of a single number that evaluates the efficiency of a system to reduce noise transmission through the air. The higher the Sound Transmission Class (STC) rating, the better the system is designed. Generally, an increase of 10 points in STC equals a halving in perceived sound. If the building design involves the location of offices or rooms nearby, it is recommended that felt and thick rugs with a rubber bottom layer be used, which may help to mitigate the impact of footsteps. The standard measure that should be consulted for sound transmission by shock is ASTM E492. The classification consists of a number of sound energy reductions. The higher the Impact Insulation Class (IIC), the better the system is designed. Indications from Middleton et al. [6] are highly recommended for technical aspects and design criteria related to the effect of vibrations from floors with elevated movement of people and use of equipment.

Other actions to eliminate acoustic noise and vibration include structurally disconnecting floors and ceilings with resilient subfloor and false – ceiling systems with insulation, as well as insulating plumbing and electrical duct structures with felt and resistant hangers. It is recommended that standard ASTM E497, regarding the installation of lightweight soundproofing separations, be consulted and followed. As a general recommendation, all the elements used in the precision – microscope room(s) must comply with the standards on sound – absorbing materials for construction materials, according to standard ASTM C423. This classification denotes the ability of a material to absorb sound waves instead of reflecting them. The higher the Noise Reduction Coefficient (NRC), the better the material is to absorb sound energy. Finally, as for the limit values each microscope may differ in the amount of allowable acoustic noise and vibration. For instance, in the case of acoustic vibrations, in its Tecnai F30® and F20® microscopes, FEI™ recommends less than 70dB of environmental noise for the full spectrum and less than 55dB for the third octave band between 10Hz y 1kHz (In this case the octaves begin at 10Hz, 20 … up to less than 80Hz) [7].

Vibration: According to the general IETS (Institute of Environmental Sciences and Technology) and ASHRAE (American Society of Heating, Refrigerating and Air-Conditioning) criteria, sensitivity levels of vibration that may be present for a bench binocular microscope zones are 100µm/s, with equipment whose resolutions range from 40X to 100X. In the same line, 50µm/s for laboratories that use microscopes with resolutions that range up to 400X, and 12.4µm/s for microscopes with a capability of 1000X [8]. The above levels differ greatly from the case of TEM, which – while being very sensitive to LFV – could have different limits in the permissible amount of vibration depending on the manufacturer (<<12.4µm/s). As general information, the higher and more slender the microscope column, greater is the balancing triggered by LFVs. According to Muller et al. [5], most shock – absorbing vibration systems do a good job above 100Hz; however, depending on the resonance frequency, they can even amplify the low – frequency effect (1-10Hz).

At the policy level, generic criteria have been used to define vibration levels in scientific and metrology buildings. In 1982, the Ungar & Gordon [9] criteria, originally known as broadband noise (BBN) criteria and closely associated with the microelectronics and optoelectronics industry of the time, were introduced. These criteria were subsequently reported as standards by IEST. In the early 90s, the United States National Institute of Standards and Technology developed criteria, generically known as NIST-A, which have gained popularity in the nanotechnology community worldwide [10]. The NIST-A standards established vibration levels of 0.025µm/s in the range of 1-20Hz and 3.1µm/s between 20-100Hz [11].

Sensitivity to LFV of nanoscale – level metrology equipment can become a problem since many buildings manifest natural vibration frequencies in the order of 5 to 10Hz. Alternatively, the LFVs of less than 5Hz, which may well come from the foundations of the building or from external sources, are commonly minimized by placing the microscope only on a concrete isolated slab or a floating floor (Fig. 1). The planning and design of this alternative, after constructing the building, may be much less expensive than using vibration – compensation systems, which, however, may operate well below 10Hz in some cases [3].

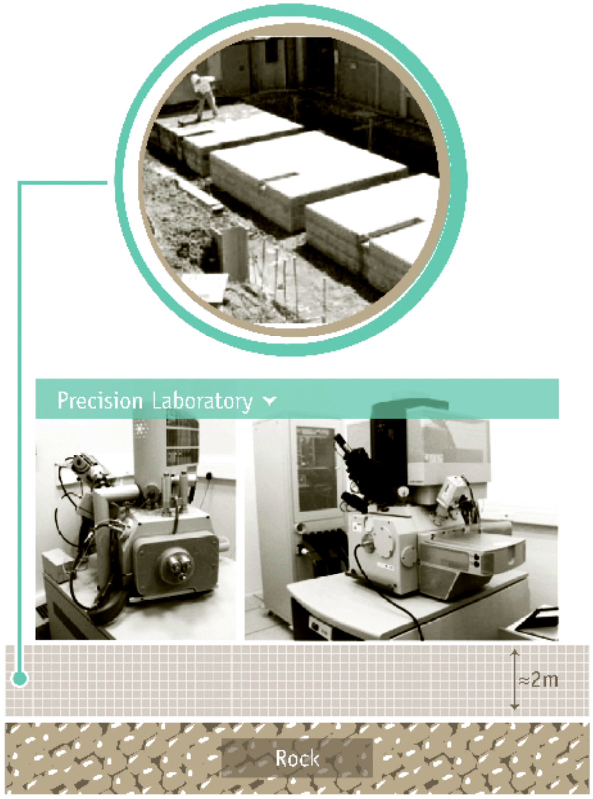

Fig. 1. Situation of concrete platforms in precision laboratories to attenuate LFVs (The height of the concrete platforms depends on the depth of the location of the bedrock and other design aspects to ensure the lowest LFV level)

As for vibrations at higher frequencies (30, 60 and 120Hz), such as those generated by AC engines, they prove to be equally attenuated in the environment of the instrument as long as floating slabs are implemented. To implement floating slabs or floors, spaces are previously arranged and conditioned. Then, depending on the number of instruments, the slabs are placed with a suitable level of separation (2-3cm approx.) from the building walls. In this separation space, polystyrene plates can be placed later on, without necessarily being forced between walls, so that they continue mitigating the absorption of low – frequency sound, as well as being useful to prevent instruments (keys or tools) from falling between the slab and the wall. As a reference, the dimensions of cubic concrete platforms could be 1m x 4 x 4m (~30 to 34ton). As a matter of comparison the average weight of a TEM microscope in 1500 kg.

Once the concrete slabs have been implemented, the maximum level of attenuation achieved through programmed testing must be verified. The use of anti – vibration slabs must seek to ensure much less than 1μm/s in the range of low frequencies (1Hz < λ < 10Hz). It is worth noting that the type of concrete used in these cases is special in its characteristics of absorbing vibrations. H. Amick et al. [10] have reported different mechanisms to increase the capacity of the concrete to suppress vibrations. Some of these mechanisms include the use of composite materials and polymer concrete mixtures [12], [13].

The structural design of the building usually precedes the selection of the final precision instrument. For that reason the engineer or architect usually uses generic criteria (*i.e.* NIST-A) in order to define the mechanisms and correct the potential vibration phenomena. However, these criteria and manufacturer's specifications commonly use terms that are not similar. In these cases, for the terminology, metric and conversion methods, it is recommended that users consult Salyards and Firman [12], who have compared the maximum levels of vibration reported by different manufactures for SEM and TEM devices. This guide is useful to interpret most suitably the majority of the terms suggested by the potential manufacturer of the instrument.

Finally, in the case of new buildings, due to phenomena of settling, an increase in the number of persons and the increasing use of spaces, it is advisable to perform routine measurement tests of the vibration level in the precision laboratory(ies) [13]. These measurements are also required to ensure the useful life of the equipment, reproducibility of results, maintenance of certification, recognition and demand for laboratory services. It is recommended having manual equipment for the recurrent assessment of the vibration level in precision areas. Equipment for this purpose may be portable meters or accelerometers, such as Fluke-810®, Wilcox-Spicer Instruments® or Brüel & Kjær® 2250H, 2515, among many other available brands

Electromagnetic Interference: The main sources of AC that produce electromagnetic interference (EMI) are unbalanced electrical charges. On the other hand, there is actually little to do in the case of DC sources or fields, such as those from nearby elevators, buses or electric trains [3]. For an ideal conductor, the magnetic field B (mG) is given by Ampere's law (Ec. 1), where l (mA) is the current in the conductor and d (m) is its length:

$$B=2l/d \qquad (1)$$

As a practical reference, a field of 0.3mG rms can be detected in an STEM image with a resolution of 0.3nm. Thus, less than 0.1-0.2mG rms are required in order to obtain a clear image in the STEM mode with a resolution of 0.2nm. Equation (1) puts this in evidence: in 1m separation, from a conductor carrying only 0.5mA, the magnetic field will reach 1mG, enough to affect the performance of a microscope in the STEM mode. This does not necessarily mean that lighting a 60W light bulb causes all the images in the STEM mode to be blurred; however, the magnitude of an incandescent light bulb, even when the magnetic field is small may become perceptible.

In general, to minimize the EMIs and particularly since low – frequency radiation (1-3kHz) is less effectively shielded [3], all wiring, including lighting, should be located as far as possible from the microscope column, seeking a magnetic – field radiation intensity from electric transmission of less than ~0.1 μG a 60Hz (0 -2.5 mG →Low, 2.5 - 7.0 mG → Medium, >7.1 mG → High). As an example, an Aberration-Corrected Electron Microscope (ACEM), with possibilities to generate sub-Ångström-level images, requires environments whose electromagnetic fields are well below 0.3 mG [14]. As a complementary example, in the installations of the IBM nanotechnology laboratory in Zurich, magnetic fields guarantee less than 0.05 mG (The Earth's magnetic field is 10,000 times higher ~0.5mG). Similarly, in the case of the SEM microscopy, the EMIs are the most common cause of the geometric distortions present in high – resolution images [15].

Subsequent, all power and signal cables, as well as all coolant hoses, should be channeled into special tubing. It is recommended using STP (shielded) – type or ScTP – FPT (overall shielded or unshielded) twisted pair cable, which, although more expensive, allows environments free from electromagnetic and radio – frequency interference, as well as avoiding the Crosstalk effect, whereby a transfer occurs from one signal pair to another nearby. The twisted longitude ranges from 5 – 15cm, while the smaller the twisted longitude is, greater is the quality of the cable. It is also recommended that support networks for the precision rooms be designed and mounted according to the TIA/EIA-568-B standards for commercial wiring telecommunication products and services, and the other characteristics for Category – 5 structures cabling. (*i.e*. CO-NETIC® twisted magnas and INTER-8® woven wire).

Other effects of electrical interference and vibration are controlled by incorporating dielectric materials needed to eliminate electrical conduction pathways in all fire ducts and networks, cooling – water lines, hot water and sewers (i.e. Nanovate™). Also, a wiring distribution should also be ensured around the entire precision laboratory (ies) (ring distribution), so that the only thing that should be introduced into the microscope room is those lines that are necessary for normal equipment operation. At the construction level, in order to reduce the impact of electromagnetic interference, using concrete blocks – instead of steel rods – on every wall of the precision room(s) is recommended, in order to eliminate possible magnetic fields generated by currents that can travel through the metal.

The lighting design also becomes relevant. Incandescent light bulbs are used instead of florescent lighting to mitigate the EMI. However, even when the EMI from an incandescent light are certainly lower compared to compact fluorescent lamps or fluorescent tubes, both types of lighting differ in their levels of electromagnetic emission. For this reason, it is important to have plenty of space and high ceilings and place lighting as far away as possible from the microscope column, which reduces the $1/d$ factor in Equation (1). Faraday cages also protects against static electric fields, as well as placing the main electrical transformers and equipment at opposite ends of the building where the precision instruments are located.

In some laboratories dedicated to STEM, shielding systems against electromagnetic interference, manufactured with MuMetal® (a metal – alloy trademark developed by *Magnetic Shield Corp.* with magnetic – shielding properties) are used. The magnetic shields works by generating a pattern of low reluctance to external fields, while slowly varying magnetic fields are not greatly attenuated. Therefore, the shielding systems are *only effective if the entire instrument room is enclosed or completely shielded* (6 sides), which clearly increases installation costs. Additionally, walls shielded in a material, such as MuMetal®, need to be very rigid since it is otherwise possible to generate acoustic and mechanical – vibration coupling mechanisms with magnetic fields of equal frequency [5]. As a rule of thumb according to D. A. Muller, the magnetic field can penetrate approximately five times the size of any holy or unshielded area [3]. This emphasizes why it is important to have large spaces and high ceilings in the site where the microscope is located (Eq. 1). Additionally, any field inside a narrow room can be reflected like a mirror in the relatively close shielded walls. For these reasons, the choice of a room with ample installations is comparatively a more – economic choice.

Another parallel measure is the shielding against local eddy current or Foucault currents, generated in a conductive mass flow by varying the magnetic induction.

In this case, the fields are attenuated so that the shielding need not be continuous. The thickness of the shielding material is determined (in meters) by the depth of penetration (δ) in Eq. (2). In this equation when a conductor is subjected to the action of electromagnetic waves, a current, whose density decreases with the distance inward from the surface of the conductor, circulated through it.

$$\delta = \sqrt{[2/(\sigma\mu\omega)]} \quad (2)$$

σ: Conductivity, μ: Magnetic permeability, ω: Frequency of incident radiation

An incident electromagnetic wave in a highly conductive medium can be exponentially attenuated as a function of δ. Nevertheless, commonly useful materials for electromagnetic shielding are also good conductors. Copper, which is usually expensive, guarantees a δ~ 2μm, at a frequency of 1GHz. Meanwhile, aluminum, in which δ~ 5mm a 60Hz, guarantees a 1cm linear thickness attenuates an external field by a factor of ~7 and 2cm by a factor of x50 [5]. Likewise, the dependence of δ with $1/\sqrt{\omega}$ further emphasizes the sense of shielding for high frequencies, while being almost useless in low frequencies or quasi-DC disturbances (such as those from nearby elevators, buses or electric trains [3]). In the latter case, a parallel method to shield from Foucault currents is the active cancellation through broadband sensors, which can better compensate the fields from distant sources. Because a magnetic field is a vectorial magnitude, spatially defined by its direction and magnitude, the basic principle of active cancellation consists in meeting a force of equal intensity and opposite direction, with the result being zero at that point of space.

As previously mentioned, the general recommendation is to place the precision rooms on the ground floor of the building [16] minimizing some of the adverse environmental effects that affect the performance of the instruments. In this sense, a less obvious aspect is related to the presence of moisture, either on walls or ceilings, as a result of rain or even melted snow that can be filtrated. This latter aspect is clearly not subject to analysis in countries where there are no seasonal changes. Although these situations should be anticipated and properly corrected, the potentially generated magnetic field can be approximated using Eq. 1 and assuming an ideal conductor surrounding the place, with the capability of transporting up to 100A of AC [3]. Especially in the case of new buildings, due to the progressive use of spaces, routine testing to assess the level of electromagnetic interference is recommended. These measurements are required to ensure the reproducibility of results, maintain potential laboratory certification, its recognition and a high demand for services. It is recommend having portable guassimeters, with a frequency range between 0-300Hz and sensitivity of <<0.1mG rms (i.e. cole-parmer® and technitool®, among other brands).

Moreover, for the case of characterization studies or work involving automated data – acquisition sessions, it is very important to have a comprehensive control room (Fig. 2), from which the remote operation of equipment is permitted. Parallel to this, while the equipment is operating, it is possible to interrupt the illumination of the room and operate from the control room, thus decreasing the potential EMI caused by the lighting [17]. A third advantage is related to the possibility of isolating heat sources (people and computers), because as will be mentioned below, heat generation interferes with the proper performance of the instrument. Besides the advantages already mentioned, the option of an overall control room is of special interest for new buildings, where more than one precision microscope or instrument could be installed over time. Additionally, the preliminary design of a new building permits defining the best distribution of electrical and telecommunication networks between the control and precision rooms (Fig. 2).

Finally, it is worth noting that many peripherals, especially those of a certain age, can generate important AC fields. These types of peripherals, such as small black – and – white monitors, can generate fields of up to 60mG. Even computer monitors with TCO-95 certification can generate up to 10mG. For this reason, even though they are more expensive, it is advisable to use LCD screens, which do not possess magnetic coils and generate at most 0.1mG at distances of a few centimeters [3]. A TCO certification is also recommended, which is the most important demanding standard in the world for computer and telecommunication products that combine ecological qualities and high – performance design.

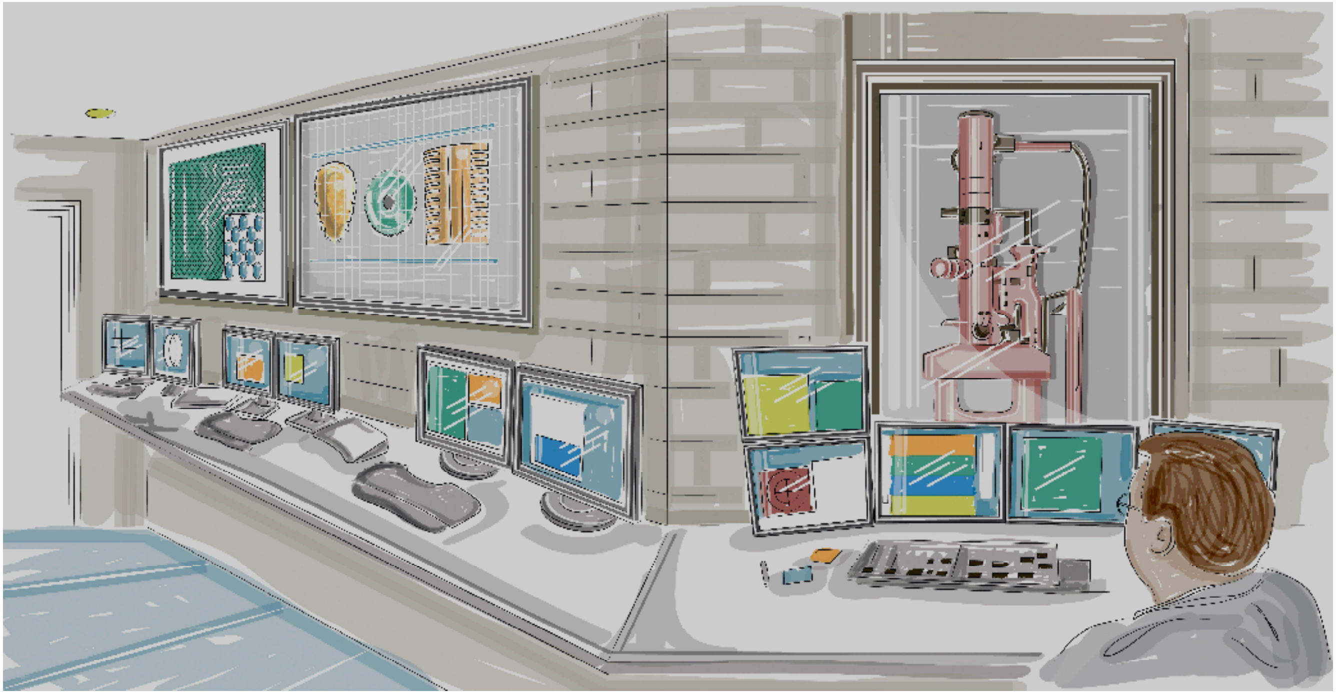

Fig. 2. Scheme of microscope control room

Barometric Air Pressure and Temperature Control: The air – management systems (heating, ventilation and air conditioning) should be designed so that they supply just the right amount of cold air, which also permits balancing the amount of heat generated (Eq. 3) from different sources (including the technical operator and probably another person to study special samples) and ensures uniform temperatures in the microscope room(s), with variations generally close to 0.1°C/h and a maximum of 0.2°C/h [14].

$$q = mC_p\Delta T \qquad (3)$$

q: Heat generated, C_p: Heat capacity of the air, m: Mass of air and ΔT: Temperature gradient between input and output.

Imbalances in the amount of heat generated, as well as fluctuations in the temperature and barometric pressure of the microscopy room as a function of the air that circulates close to the microscope column, can generate displacements in the image, especially in acquisition periods of 1.30s. In the STEM mode, the effects are more noticeable at low frequencies (< 10 Hz). Also, the air circulating around the column generates phenomena of thermal fluctuation and acoustic vibration that are closely related. As previously explained, the microscopes are generally very sensitive to LFV. In this case, the resonance frequency of the anti – vibration isolations systems of the instrument are especially sensitive to air movement. As example, the generation of noise in the instrument room of at most 50dB, as a result of operating the air – conditioning systems, may generate a displacement of the column of up to 0.3Å; 40dB generated a displacement of ~0.1Å [5]. Table 1 lists the recommended noise values for most Transmission Electron Microscopes.

Table 1. Levels of sound intensity

dB	Examples
200	An atomic bomb or similar
180	Volcano explosion / rocket at takeoff
140	Pain threshold
130	Plane at takeoff
120	Airplane engine running
110	Concert
100	Electric Perforator
90	Traffic/ fight between two people
80	Train
70	Vacuum cleaner
50/60	Agglomeration of people / dishwasher
<50dB	Recommended value for most Transmission Electron Microscopes
40	Conversation
20	Library
10	Quiet breathing
0	Hearing threshold

Complementary, a second roof, distant from the ground, made of flexible polyurethane foam with a superficial anecoide finishing could be used in order to decrease the noise interference. Anechoic chamber or anecoid room can also be specially designed to absorb sound that falls on the walls, floor and ceiling of the precision room itself. Although it is understood as an egg – box structure, it should not be assumed to be equal. Particularly, anecoid shapes are much more effective to attenuate sound. As an economical alternative compared to anecoid foam, fiberglass is often used to muffle sound in large spaces and has a good performance in relation to the attenuation of low – frequency sounds.

Based on the above, air currents should be minimized, for example, by arranging the air inlets to the precision room along the sides farthest from the microscope column, so as to provide a laminar flow in both instrument surfaces (column) and on the floor. Also a descending air – flow system could be implemented [17]. According to Muller the maximum air velocity should be less than 7.62cm/s, for a resolution of 0.2nm in the STEM mode [2]. L Brown recommends less than 5cm/s [14]. As a comparative datum, the average value of air speed for people walking is ~178cm/s. To provide a suitable temperature and adjust the airflow, frequency inverters are also used in order to allow the maximum airflow, according to the design (normally ~960cfm) and generate less than 5cm/s of vertical speed. In general, 500cfm with approximately 6 cycles/hour is more than enough to control the temperature at values lower than those required by the design, further minimizing air velocity in the precision room.

On the other hand, in the precision microscopes, the sample – holder system is generally a goniometer that allows three translations (x, y, z) on the sample, as well as three rotations known as Euler angles (α, β, γ). With this range of movements it is possible to vary the position of the sample relative to the incident rays as desired. If the sample is a single network (monocrystal), this mechanism can observe the diffraction associated with this network from any direction relative to the direct beam and the crystallographic axes of the material. The goniometer is very sensitive to changes in pressure. Typically, the sample is placed under vacuum conditions (~$3x10^{-6}$ Pa), while the other end of the sample – holder system is maintained at atmospheric pressure (~ 10^5 Pa), so that small variations – a few Pascals – in the barometric pressure of the room can generate shifts in the images obtained. D. A. Muller et al. report that changes of 1Pa in the pressure can generate deflections of nearly 0.1nm [2]. In this sense, the building design must consider the potential variation of pressure when the doors are closed or opened, not only to the precision room but as well as the main access areas of the building. It is also important to be careful in selecting equipment, so that the microscope can have suitable mechanisms for the transfer of samples, control of the pressure in the sample – holder system and their eucentric positioning. This means that for any angular movement that is applied, the sample does not undergo translational – only rotational – movements. The sample will behave as it were in the center of an imagery sphere defined by the Euler angles of the goniometer.

According to this, the barometric pressure of the room where the microscope(s) are located is generally controlled at the same time by sets of double doors (air locks), so that the pressure inside the room is greater than it is outside, thus preventing the entrance of dehumidified air into the room. The plastic layer mentioned in the paragraph on Acoustic Noise, used around the entire perimeter of the precision room(s), is also implemented to prevent mitigation of dehumidified air at a different pressure, to and from other common areas.

As an example, Muller et al. [5] illustrate a STEM microscope room in which, unlike a conventional air – flow system, air conditioning is supplied through a duct furnished with a porous mesh, thereby ensuring a laminar flow. Radiant cooling panels (RCP), inside of which water circulates to condition the room temperature, can also be observed. The STC classification of these radiant panels is very low (when filled with water), which is why different panels to attenuate sound (up to ~15cm from the wall) are often placed parallel to the walls, thus improving the absorption of low – frequency sound. Since most air conditions systems operate under convection mechanisms to remove heat, the use of RCPs, because of their mode of operation, allow convection systems to slow down and, at most, be used to control room humidity [5]. A second advantage of the RCP systems related to temperature stability compared with convective conditioning systems. In any case, in order to have energy – efficient buildings and to emphasize projects that intend to apply LEED™ certifications, it is necessary to perform the calculations and comparatively establish the potential of either alternative.

As a general and complementary recommendation, to prevent temperature variations in function of the cycling needed for the air, it is recommended that the microscope rooms be generous in space (having impellers for the heating, ventilation and air – conditioning systems), as well as placing the least amount of heat sources close to the instrument. Indeed, most of the heat generated (~75%) can come from the electronic cabinets and power sources required from the microscope. This, together with the effects of acoustic vibration and possible electromagnetic interference mentioned above explains why it is common practice to have a spare room where the instrument electronics and other support equipment are placed.

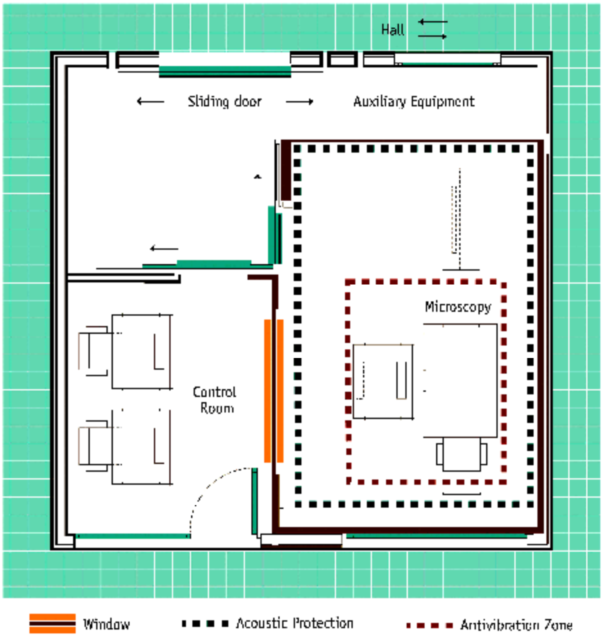

Fig. 3. General scheme of a typical laboratory to operate high – resolution metrology and microscopy equipment

Fig. 3 illustrates the general surface of a nanoscale metrology laboratory for TEM. This surface can be adjusted according to need. The minimum values of the area necessary for equipment are approximately 4m x 4.27m (~17.08m^2) as useful area; plus 1.22m x 4.27m (~5.20m^2) approx. for a spare room, which houses the instrument electronics and other support equipment. Especially because of the progressive use of spaces, it is advisable to perform routine measurements of air speed in the laboratory. These measurements, which can be performed using thermal anemometers with adequate resolution, are required to ensure the quality of the images generated in the microscope, as well as possible thermal fluctuations [14].

Final General Recommendations. On a technical level, among last aspects, the author recommends: Using STP (shielded) twisted pair cable for the support networks that will supply the precision rooms, considering the TIA/EIA-568-B standards for channeling power and signal cables, as well as cooling – water hoses, in special tubing.

Ensuring that all pipes and fire networks, cooling – water lines, hot water, sewers and the like, that directly go into the microscope room(s), incorporate dielectric materials needed to eliminate electrical conduction pathways.

Placing transformers and main electrical equipment on opposite ends of the building where the precision instruments are to be located.

Ensuring that all instruments comply with sound–absorption standards for building materials, considering the ASTM C423, ASTM E497, ASTM E492 and ASTM E90 standards. A noise intensity of much less than <50dB (<60Hz) inside the room(s) once the equipment has been installed and under normal operating conditions (i.e., in permanent housing conditions) must be ensured.

Using effective means to control and eliminate high– and fundamentally LFV that could reach the instruments. The selection and design of alternative solutions, such as a concrete buffer for the location of each microscope, should be performed according to the specific local conditions of the area, the probable vibration levels in the future and clearly, according to the instrument manufacturer's specifications.

In designing the spaces for microscope and precision instruments, ample, high – ceiling spaces should be provided, as well as placing lighting as far away as possible from the area where the microscopy column(s) will be located. The use of anti – vibration slabs ensure less than 1μm/s in an interval of low frequencies (λ): 1Hz < λ < 10Hz. The planning and design of this alternative, before constructing the building, is much more economical than using active systems of vibration compensation.

Having one or more independent rooms for equipment control, so that heat–generating sources, (including people and computers), can be isolated. Although it is more expensive, it is recommended using LCD screens, which do not have magnetic coils and generate at most 0.1mG at distances of a few centimeters. Also, it is advisable to have one or more support rooms, where the instrument electronics and other support equipment will be placed.

Designing air–management systems to ensure uniform temperatures, with maximum variations of 0.1 - 0.2°C/h in the room(s).

Having air inlets into the precision rooms along the sides farthest from the location of the microscope column and providing a laminar flow, with a maximum air speed inside the room, mainly in the area where the microscope column will be located, of less than 5cm/s. Finally, placing the laboratories on the ground floor, or in the basement of the building, with no filtration or moistures.

CONCLUSIONS

Facilities are truly a part of the microscope and must be budgeted together (Fig. 4). Especially in the case of new nanoscopy laboratories, due to the potential phenomena of settling, a progressive increase in the number of persons and the increase use of spaces, it is recommended that routine measurement testing be conducted about the vibration levels.

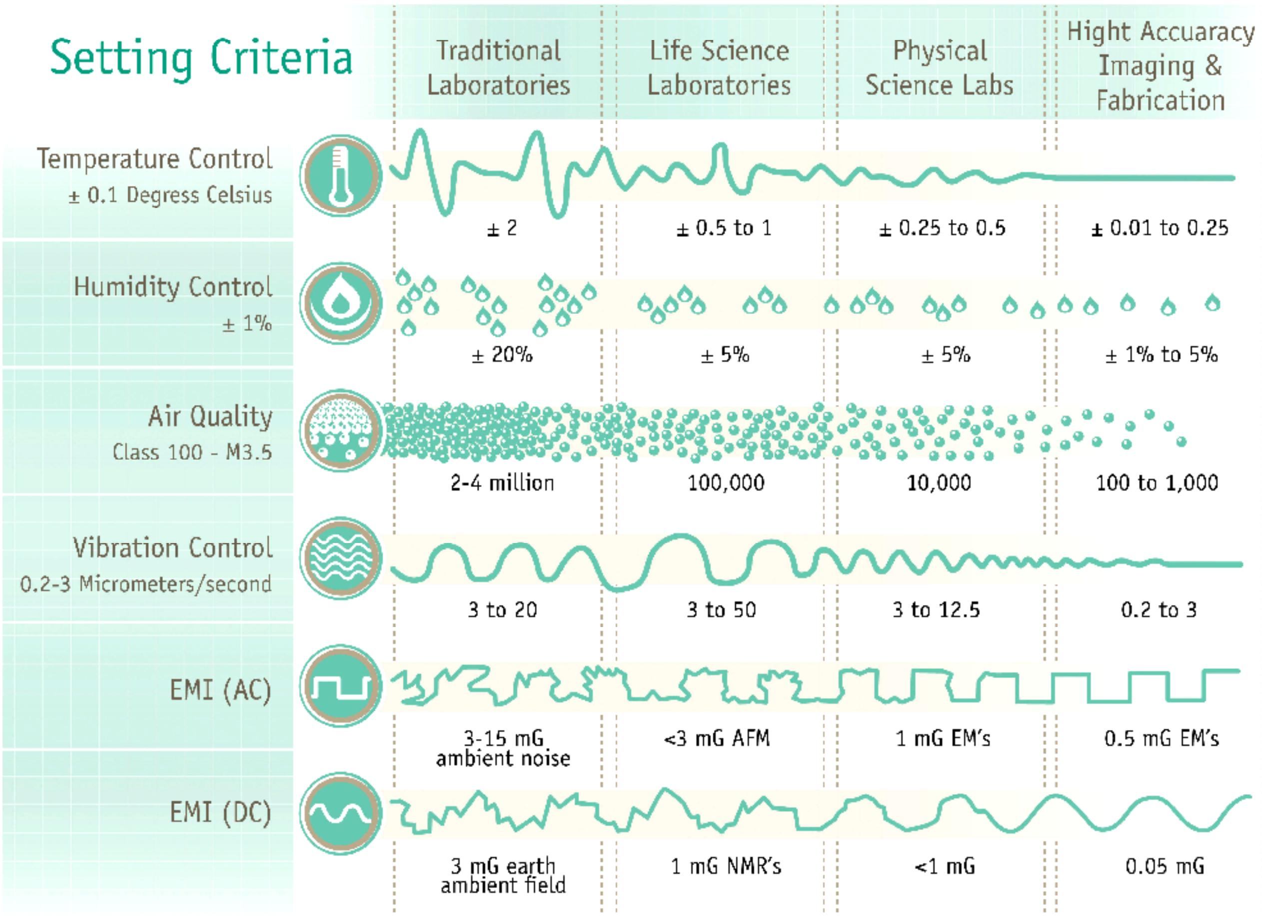

Fig. 4. General Setting Criteria for Electron Microscopy Laboratories

These measurements will also be required to guarantee the useful life of the equipment, reproducibility of results, maintenance of certification, as well as recognition and demand for laboratory services. Thus, it is recommended having manual equipment for the recurrent assessment of the vibration level in the precision areas. Equipment for these purposes may be portable gauges or accelerometers.

In general, to minimize EMIs, all wiring – including lighting – should be located as far as possible from the microscope column, aiming for a radiation intensity of the electromagnetic field through electric transmission at less than ~0.1mG at 60Hz. This creates possibilities to obtain images at the sub-Ångström scale.

It would be ideal to provide high – quality services in advanced microscopy if the design parameters set for the building allow ensuring a magnetic – field threshold of less than 0.05mG.

The design of zones for microscopy and precision instruments requires spacious, high – ceiling installations. This allows minimizing the influence of effects associated with acoustic vibration, possible electromagnetic interference, temperature variations in function of the cycling necessary for air, as well as having less heat – generating sources near the instrument(s). The overall surface of a nanoscale metrology laboratory can be adjusted, depending on the need.

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