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Nanometrology Room Design: The Performance and Characterization of the Kevin G. Hall High-Accuracy Laboratory

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Abstract

New buildings focused on the practice of nanotechnology reflect a pressing need to develop advanced techniques to enable reliable work at the nanoscale. Often when planning a nanotechnology building, a decision must be made to include high-accuracy nanometrology rooms. The purpose of these rooms is to provide high-quality space that can be utilized on a daily basis to facilitate experiments requiring nanoscale precision, to develop new instrumentation, and to develop new techniques capable of probing the nanoscale. Typically, these rooms reduce vibration (including acoustic noise) and electromagnetic interference to very low levels while maintaining a high level of temperature stability. This study describes the characterization and performance of the Kevin G. Hall Nanometrology Laboratory located in the Birck Nanotechnology Center at Purdue University.

KEYWORDS

Nanotechnology, nanoscale, nanometrology, laboratory design, scanning probe microscope

INTRODUCTION

Developed nations are constantly striving to establish and maintain an advantage in new and rapidly emerging technologies. This competitive quest for technological preeminence requires the construction of world-class research facilities, the education and training of next-generation students, and the creation of a knowledge base that will fuel economic development for the next 50 years. Today, these efforts are focused on the development of nanoscale science and technology, driven by the realization that properties of materials with nanoscale dimensions differ in fundamentally important ways from those properties appropriate for materials at the macroscale.

Concurrent with the rise of nano-based technology is the need for new and innovative measurement techniques that provide a reliable metric at the nanoscale. It is widely believed that standards in nanotechnology will develop from a quantum metrology that will replace traditional material standards with more precise quantum standards that will be accurate to limits determined by the laws of quantum mechanics.¹ In the broadest of terms, nanometrology can be defined as the accurate measurement of physical properties of a material that has at least one dimension smaller than 1 μm (39.4 $\mu\text{in.}$). Activities in this arena must clearly range beyond geometric length characterization alone. Often, measurement principles that work at the macroscale become untenable at the nanometer scale, requiring the invention of new measurement techniques and protocols. Continued advances in precision engineering, optics, electronics, materials, and molecular biology will increasingly require high-accuracy qualification of mechanical and electrical properties in addition to physical dimensions.^{2,3,4}

For geometric characterization, atomically resolved crystalline surfaces will serve as the new standards of length and angle at the nanoscale. The rapid placement of features on substrates will require geometric accuracy in the submicron range. To become commercially viable, fabrication and assembly at the nanoscale will likely become independent of the expensive infrastructure

developed for cleanroom manufacturing protocols. A key role will be played by quantitative scanning probe microscope (SPM) techniques.

Improved methods for characterizing nanoscale processes require new metrics to ensure reliability and repeatability. To accelerate these trends, national agencies have been established to develop uniform practices and standards at the nanoscale. This worldwide effort is remarkable because of its breadth, with all industrialized countries now having their own dedicated agencies with a mission to advance nanoscale metrology: China (NIM), Denmark (DFM), France (LNE), Germany (PTB), Italy (IMGC), Japan (NMIJ), Korea (KRISS), Netherlands (Nmi-VSL), Poland (GUM), Russia (VNIIM), Spain (CEM), Switzerland (METAS), Taiwan (CMS), the United Kingdom (NPL), and the United States (NIST).

The upswing of nanomanufacturing efforts within the past five years is also a notable trend. Industry may require high-quality nanometrology rooms for the robust and repeatable nanofabrication of structures with atomic control of size, shape, spatial position, and chemical composition. Examples include IBM's 1 Gbit/mm² (1 terabit/in.²) Millipede project,⁵ the Molecular Measuring Machine,⁶ the mass production of repeatable patterns using dip-pen lithography,⁷ and the use of nanomachining for geometry reconstruction to repair defects in photomasks using scanning probe microscope techniques.⁸

An enabling step in these worldwide efforts is the design and construction of high-accuracy nanometrology laboratories. Because of the key role played by such laboratories, it is worthwhile to document what can be achieved in the planning, design, characterization, and performance of nanometrology labs. By learning from prior efforts, the performance of future nanometrology rooms can be further improved.

For these reasons, the authors discuss the characterization and performance of the Kevin G. Hall Nanometrology Laboratory, located in the Birck Nanotechnology Center at Purdue University.

THE DESIGN OF A NANOMETROLOGY LABORATORY

In general, research laboratories are moving from older buildings, designed and constructed 20–40 years ago, into newer buildings that emphasize recent advances in research and development. This trend is likely to accelerate with advances in nanotechnology.

New laboratories designed to enable nanotechnology provide advanced measurement capabilities that are nonexistent in previous-generation laboratory space. For the purpose of this article, such space will be referred to as high-accuracy nanometrology rooms. Figure 1 illustrates the need to separate such laboratory space from unwanted environmental influences.

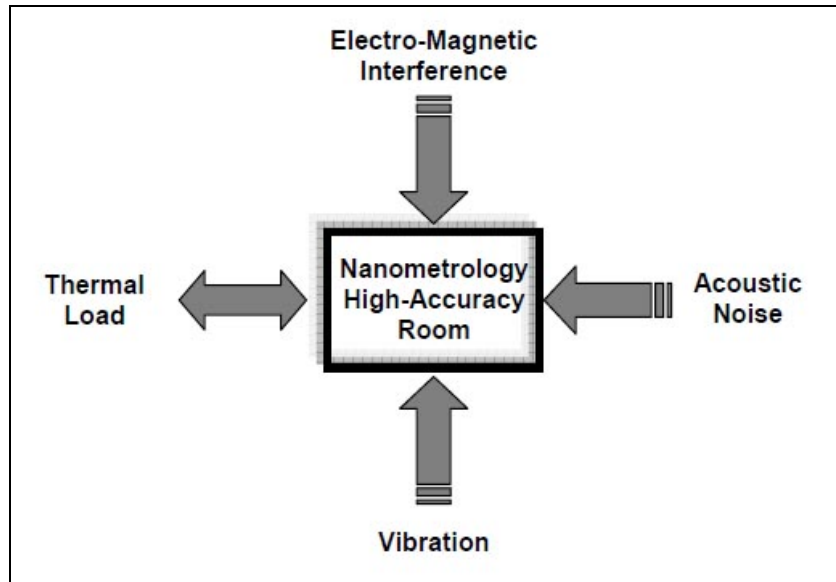


Figure 1. A schematic diagram illustrating environmental sources that may limit the performance of a high-accuracy nanometrology laboratory.

It is easy to discuss hypothetically the desired characteristics of a nanometrology laboratory. A nanometrology lab should be located far away from roads, parking lots, elevators, and air-handling equipment to mitigate ground-borne propagation of transient vibrations from automobile and railway traffic, construction equipment, blowers and pumps, etc. Such a lab should also be isolated from the structural supports of the main building as much as possible. Ideally, the laboratory layout should effortlessly provide restricted access. Such a lab might be buried underground to facilitate a constant temperature and be equipped with an adjacent control room that houses much of the electronic instrumentation. In general, air handling should be designed to prevent building air from blowing directly on equipment. Humidity should be controlled and air quality, though not generally required to be of cleanroom quality, should be filtered to reduce particle concentrations by roughly a factor of 10 below that of air circulating throughout the building. Support structures for sensitive equipment should be isolated from surrounding floors and designed to damp unwanted vibrations. The lab should be far removed from high-voltage transformers and high-current electrical power lines. Independent and distributed, quiet electrical grounds should be available throughout the lab. Any required 110-V power lines should be filtered, regulated, and distributed by twisted wires to reduce stray magnetic fields. The use of discharge lighting (and ballasts) should be reduced to a minimum. The temperature and humidity of the room should be carefully regulated. The room should be electrically shielded to reduce the influence of outside electromagnetic (EM) radiation. Sound-absorbent walls, ceilings, and floors should be incorporated to reduce background acoustic noise to a minimum.

Many of these desired characteristics have been incorporated in the Kevin G. Hall Nanometrology Laboratory in the Birck Nanotechnology Center at Purdue University, located in West Lafayette, Indiana (see Figure 2). Construction of the laboratory was completed in September 2005. The laboratory's interior dimensions are $6.7 \times 5.6 \times 3.4$ m ($22 \times 18.5 \times 11$ ft).

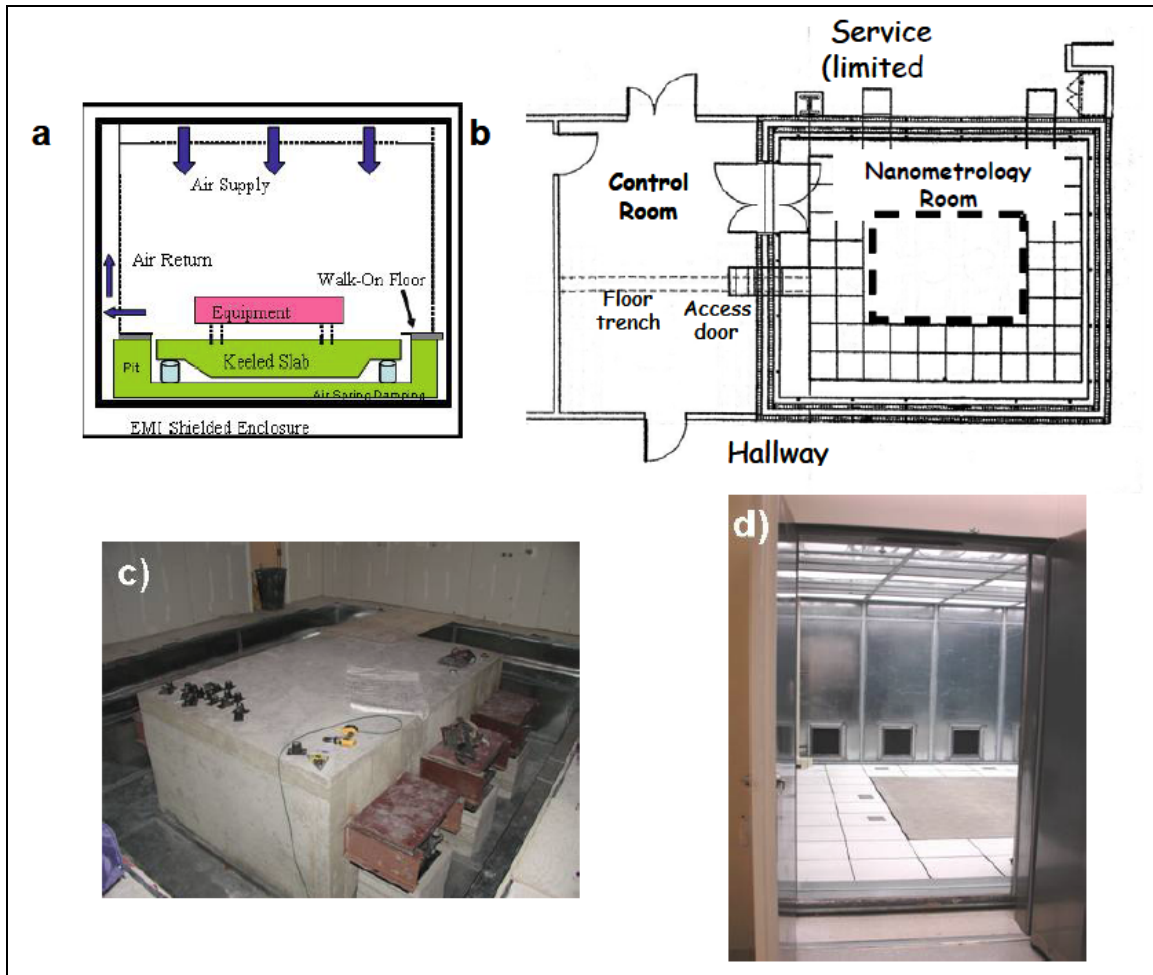


Figure 2. Overview of the Hall Laboratory. Item (a) illustrates the design of the vibration and air handling capabilities. Item (b) is a floor plan of the nanometrology room and the adjacent control room. The dashed line outlines the placement of the vibration isolation slab. Item (c) is a construction photo of the 30,000-kg (33-ton) concrete slab for vibration isolation. Item (d) is a photograph of the Hall Laboratory from the control room after construction was completed.

The following discussions summarize the performance of the Hall Laboratory and provide test results that quantify the controlled experimental environment that has been achieved.

ACOUSTIC NOISE

To characterize airborne (acoustic) noise, a measurement of the sound energy at particular frequencies is required. The sound energy can come from sources such as automobile traffic outside the building, air handlers, blowers, nearby walkways, and human conversation. For airborne noise, the frequency range of interest generally lies between ~20 Hz and ~20 kHz. Usually this frequency range is broken down into discrete segments using the well-defined 1/3-octave-band scheme. The advantage of that procedure is that the center frequencies of interest are well specified and the bandwidth about a given center frequency is well defined. The sound pressure level is often measured in dB, using a pressure of 20 μPa (2.9 nano psi) as a reference level. A root-mean-square (RMS) sound pressure level of 20 μPa at 1 kHz is often used to define the threshold for human hearing.

The acoustics of a laboratory can be classified by different noise criteria (NC) curves that specify the sound pressure levels as a function of frequency.⁹ Representative NC curves for different acoustic environments are given in Figure 3. For reference, a quiet office is typically rated between NC-30 and NC-35.

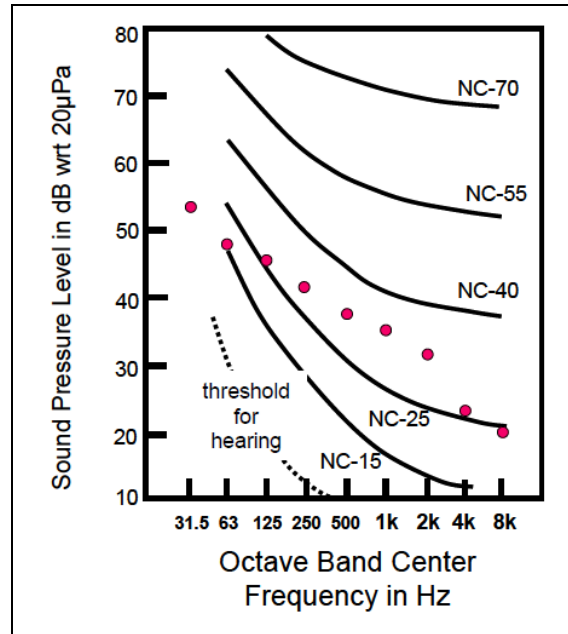


Figure 3. Data characterizing the acoustic performance of the Kevin G. Hall Laboratory. Sound pressure levels are plotted in red. For reference, various NC levels are also plotted by solid lines. From Reference 10.

The data plotted in Figure 3 represent the sound pressure level measured inside the Hall Laboratory (with respect to a reference value of 20 µPa (2.9 nano psi) at 1/3-octave-band frequencies) with the doors closed and the air-handling equipment operating. These data indicate that the laboratory warrants an NC-35 rating.

Conversational speech occurring in adjacent hallways is inaudible to people inside the laboratory. However, because the laboratory is a closed box bounded by rigid walls, the NC-35 rating is exceeded when people are speaking inside the room or when mechanically driven equipment such as a vacuum pump is energized inside the room. This acoustic degradation is related to standing-wave acoustic resonances that are set up between adjacent walls. When experiments are under way, the room is closed to humans and all unnecessary mechanical equipment is shut down. Under these circumstances, the room is well described by the NC-35 rating.

TEMPERATURE STABILITY

Temperature stability is critical in any nanometrology laboratory. For temperatures near room temperature, an aluminum rod of 1 cm (0.39 in.) in length will expand by ~ 240 nm (9.45 μm .) when subjected to a temperature increase of 1°C (1.8°F). For this reason, a high level of temperature stability is desirable in rooms designed for nanometrology.

The strategy followed in the Hall Laboratory to achieve temperature stability is indicated in Figure 2(a). The airflow to the laboratory is controlled by a separate air handler that allows computer set-point control of both temperature and humidity. An inline variable-speed motor controlled by three airflow sensors allows computer control of airflow, with adjustable airflows spanning a range of 63.7 to $255\text{ m}^3/\text{min}$ (2250 to $9000\text{ ft}^3/\text{min}$). The laboratory is equipped with four high-precision temperature probes mounted on 4.6 m (15 ft)-long electrical cables to allow placement near critical equipment. The temperature probes are initially calibrated to an accuracy of 0.0015°C (0.0027°F) with a temperature stability specification of 0.005°C (0.009°F) per year. The three set points—temperature, humidity, and airflow—are continuously monitored by a building automation system located in the building manager's office.

Airflow comprised of $\sim 85\%$ recirculating air and $\sim 15\%$ make-up air is directed into the laboratory through 12 ceiling vents. The air return to a dedicated air handler is through 16 wall registers located around the inside perimeter of the laboratory walls (three of these registers are visible in Figure 1(d)). The low wall registers circulate the return air through the laboratory's hollow walls. In this way, the temperature of the interior wall is held constant by the flow of temperature-regulated air. A similar approach has been adopted in the design of high-accuracy rooms in the Advanced Laboratories of the National Institute of Standards and Technology (NIST), located in Gaithersburg, Maryland.¹¹

The temperature stability that can be achieved by this design is illustrated in Figure 4, which plots the average temperature over a 4-day period, measured inside the laboratory by four separate thermocouples located near the upper four corners of the room. The temperature was monitored using the minimum [$63.7\text{ m}^3/\text{min}$ ($2250\text{ ft}^3/\text{min}$.)] airflow. The data clearly indicate that the temperature in the room is stable to $\pm 0.01^\circ\text{C}$ ($\pm 0.018^\circ\text{F}$). This stability is ~ 10 times better than the initial $\pm 0.1^\circ\text{C}$ ($\pm 0.18^\circ\text{F}$) design specifications for the room.

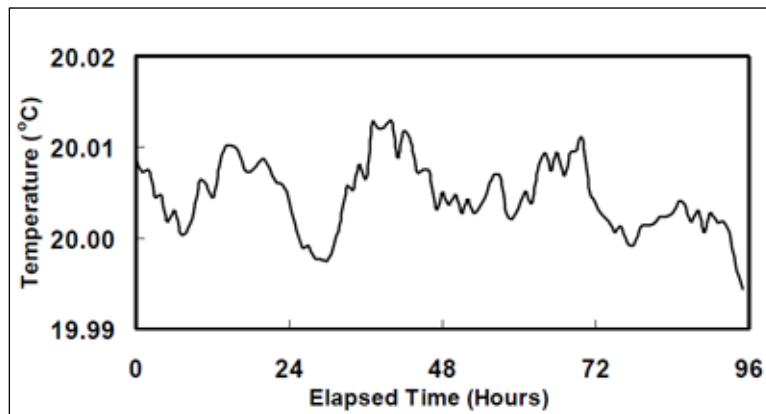


Figure 4. The results of a 4-day temperature stability test of the Hall Laboratory. Airflow was maintained at $63.7\text{ m}^3/\text{min}$ ($2250\text{ ft}^3/\text{min}$). The plotted temperature is the numerical average of the four separate temperature probe readings.

Further tests were conducted to measure the thermal time constant of the room. In these tests, the temperature of the room was allowed to equilibrate overnight before the door to the laboratory was opened. The door remained open for ~4 hours to simulate a situation in which the set-up of a new experiment might take place. Subsequently, the door was closed and the average temperature in the room was monitored to see how it returned to the set-point value of ~20 °C (68 °F). The temperature-time data, plotted in Figure 5, reveal that upon opening the door to the room, the temperature increases to near-ambient value in ~1.5 hours. After closing the door to the laboratory (at an elapsed time of ~6.5 hours), the temperature returns to within ± 0.1 °C (± 0.18 °F) of the set point in ~2 hours. Recovering the ultimate stability of the room [± 0.01 °C (± 0.018 °F)] requires an overnight (~12-hour) stabilization time (not shown).

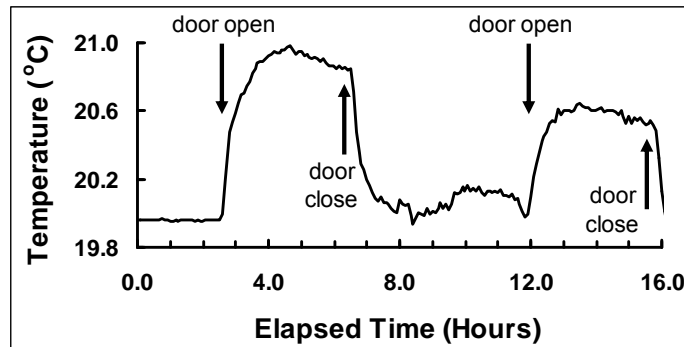


Figure 5. Temperature recovery tests conducted with an airflow of $63.7 \text{ m}^3/\text{min}$ ($2250 \text{ ft}^3/\text{min}$). The plotted temperature is the numerical average of the four separate temperature probe readings.

VIBRATION DAMPING

A high degree of vibration isolation provides an important characteristic required for any nanometrology laboratory. Typically, vibrations couple to components of sensitive equipment, causing uncontrolled internal relative motion between these components. A vibration isolation approach that is often followed requires sensitive equipment to be supported on a high mass platform designed to have a resonant frequency far below any internal resonances characteristic of the equipment itself. In addition, surrounding background vibrational noise, sometimes referred to as the “natural frequency” of a facility, should lie well above the resonance frequency of the high mass platform. In this way, the coupling of floor vibrations to equipment vibrations is drastically reduced by intrinsic coupling considerations alone. Since equipment components tend to vibrate in the 100 Hz–10 kHz range (depending on the mass and stiffness of the individual components), the high mass platform scenario requires a sufficiently large mass to lower the resonant frequency of the platform into the 1-Hz range.

In the Kevin Hall Laboratory, a concrete pit was first fabricated on grade so that the pit itself was completely isolated from the surrounding Birck Nanotechnology building. Then a 30,000-kg (33 ton) concrete slab was fabricated inside the pit (Figure 2(c)). The concrete slab (dimensions: 3.6 m (12 ft) long \times 1.8 m (6 ft) wide \times 1.4 m (4.67 ft) deep) is reinforced by steel rebar and includes three steel I-beams that protrude beyond the width of the slab. The exposed ends of the I-beams are supported by six separate air-spring inflatable isolators filled to a pressure of 0.38 MPa (~55 psi). Calculations indicate the lowest eignefrequency of this slab/air-spring system is a ~1.1 -Hz “rocking” mode that is evident when a user steps onto one end of the slab.¹²

Vibrations can be specified by either acceleration, velocity or displacement values measured at 1/3-octave-band frequencies ranging between 1 Hz and 100 Hz. Because the vibrations are assumed to be sinusoidal in nature, it is easy to derive analytical formulas for acceleration and displacement if, say, the velocity of the slab is measured.

Vibration criteria (VC) are well established in the literature and are designated by the dependence of velocity, displacement, or acceleration vs. frequency.^{13,14} In this way, the standard “vibration criterion E” (VC-E), often used for sensitive laboratory settings, specifies a constant floor velocity of 3 $\mu\text{m}/\text{sec}$ (118 $\mu\text{in.}/\text{sec}$) for frequencies $8 \text{ Hz} < f < 100 \text{ Hz}$, and a constant acceleration of 150 $\mu\text{m}/\text{sec}^2$ (5.9 $\text{m} \mu\text{in.}/\text{sec}^2$) at frequencies below 8 Hz. In general, nanometrology laboratories require more stringent vibration criteria as established by the Vibration Isolation Research Project (VIRP) authorized by NIST in the United States. VIRP led to the formulation of NIST-A and NIST-A1 vibration standards. NIST-A1 is particularly stringent and requires an RMS velocity of 3 $\mu\text{m}/\text{sec}$ (118 $\mu\text{in.}/\text{sec}$) for frequencies less than 4 Hz, and 0.75 $\mu\text{m}/\text{sec}$ (29.5 $\mu\text{in.}/\text{sec}$) velocity for frequencies $4 \text{ Hz} < f < 100 \text{ Hz}$.

After construction was complete, the vibration spectrum for the concrete slab in the laboratory was measured and the results are plotted in Figure 6. Data for both the pit floor (underneath the concrete slab) and the center of the concrete slab are presented. From these measurements, we conclude that the vibrations do not exceed VC-E at any frequency, and the slab meets the NIST-A1 criteria at all frequencies greater than 2 Hz.

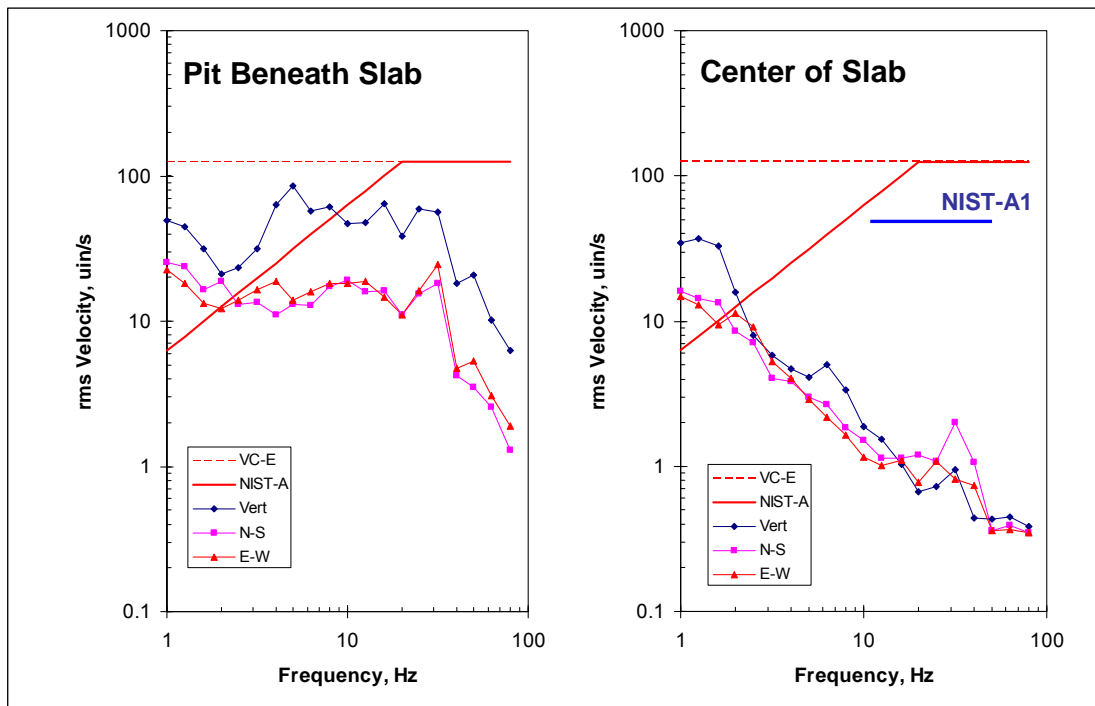


Figure 6. Vibration velocity measurements of the concrete slab in the Kevin Hall Laboratory. The graph at left shows velocity measurements as a function of frequency on the pit floor beneath the slab, and the graph at right shows measurements of velocity directly on the slab itself. Note that $0.1 \mu\text{in.}/\text{sec} \cong 0.025 \mu\text{m}/\text{sec} \cong 25 \text{ nm}/\text{sec}$. From Reference 10.

ELECTRICAL POWER AND ELECTROMAGNETIC IMPEDANCE SHIELDING

Any nanometrology laboratory requires sensitive electrical measurements and therefore must have good electrical shielding. Typical utilities directed into a nanometrology room include telephone, overhead lighting, fire alarms, internet, electrical power, and a quiet shielding ground. All of these utilities must be filtered and in some cases regulated to provide maximum low-noise measurement capabilities.

In addition, it is desirable to shield sensitive equipment from ubiquitous EM radiation emanating from cellular phones and TV and radio signals. EM shielding is determined by two mechanisms—the reflection of EM radiation from a conducting plane and the absorption of EM radiation by a conducting plane. It follows that the shielding effectiveness of a conducting plane (in dB) is the sum of the reflection loss (in dB) and the absorption loss (in dB).

Much in the same way that a transmission line reflects energy due to an impedance mismatch, reflection of EM radiation from a conducting plane is determined by a mismatch in impedance between the incident EM wave and the impedance presented by the conducting plane, which acts a reflector. While the impedance of a plane can be calculated (in ohms per square, expressed as Ω/\square) from the geometry and electrical resistivity of the metal forming the plane, it is difficult to reliably estimate the EM wave impedance. For ideal cases, such as a plane wave traveling in free space, this impedance is $377 \Omega/\square$, but for real cases, this value depends on the source of the EM radiation that is being reflected. For this reason, it is often difficult to estimate the shielding effectiveness due to reflection alone.

The EM radiation not reflected by the conducting plane is absorbed and is known to have an exponential dependence with distance z into the plane that can be specified by an exponential $e^{-\alpha z}$ where

$$\alpha = \sqrt{\frac{\pi f \mu}{\rho}}$$

(in units of m^{-1}) with f equal to the frequency (in Hz) of the EM radiation, μ is the magnetic permeability of the shielding material (in H/m), and ρ is the electrical resistivity of the shielding material (in Ωm). Once the material constants for a given shielding material are specified, the absorption attenuation constant as a function of frequency can be reliably estimated.

In the Kevin Hall Laboratory, EM shielding was accomplished by covering the entire laboratory room with two panels of $1.2 \text{ m} \times 2.4 \text{ m}$ ($4 \text{ ft} \times 8 \text{ ft}$) 11-gauge steel plate [0.32 cm (0.125 in.) thick]. The steel panels were structurally assembled on either side of a $1.2 \text{ m} \times 2.4 \text{ m} \times 1.3\text{-cm-thick}$ ($4 \text{ ft} \times 8 \text{ ft} \times 0.5\text{-in.-thick}$) plywood sheet. All seams on the interior wall of the laboratory were sealed using ironed-on copper foil or copper mesh foil (or both) placed underneath screwed-on sealing strips. To be effective, the shielding had to surround the entire laboratory, including the pit area supporting the concrete slab and the ceiling, which houses air ducts and lighting fixtures. In addition, the entrance to the laboratory from the control room was sealed by a double door (outside door to maintain temperature stability, inside door to provide EM shielding). The tightly latched inner steel door was equipped with reentrant leaf-spring seals located around the door's entire perimeter.

With this design, the laboratory met the attenuation specifications for EM radiation set out in Table 1.

Table 1. Specifications for the attenuation of EM radiation at different frequencies.

Frequency	Field	Attenuation	Attenuation Ratio
60 Hz	Magnetic	30 dB	32:1
100 kHz	Magnetic	120 dB	1,000,000:1
10 kHz	Electric	120 dB	1,000,000:1
100 kHz-18 GHz	Plane Wave	100 dB	100,000:1

Because it is difficult to effectively shield low-frequency magnetic fields, careful measurements of 60-Hz EM fields inside the laboratory were conducted using a 3-axis magnetometer. For the sake of comparison, 60-Hz measurements were performed at three locations (see Figure 2(b)): directly outside an open electrical access panel located in the service galley (worst case); inside the lab's control room (representative of the ambient 60-Hz fields); and inside the laboratory with the door closed. These measurements showed a net magnetic field of ~14 mG (1.4 μ T) outside the open electrical panel. Inside the control room, the net magnetic field at 60 Hz dropped to ~1.4 mG (0.14 μ T). When measured inside the laboratory, the net magnitude of the magnetic field at 60 Hz was below the noise level of the magnetometer instrumentation, which was ~0.05 mG (~5 nT). These values indicate that the attenuation of 60-Hz EM fields by a ratio greater than 28:1 has been achieved.

SUMMARY

The characterization and performance of the Hall Nanometrology Laboratory in the Birck Nanotechnology Center at Purdue University has been described. The laboratory meets acoustic NC-35 qualifications. After allowing ~12 hours of isolation, the temperature stability inside the laboratory has been measured to be better than ± 0.01 °C (± 0.018 °F), about a factor of 10 better than the design specifications. A 30,000-kg (66,140-lb) concrete slab provides a high level of vibration isolation that meets NIST-A1 specifications. The laboratory is well shielded from EM radiation using a double steel-plate design that surrounds the entire laboratory.

Current research under way in the laboratory involves the use of an atomic-resolution ultra-high vacuum (UHV) scanning tunneling microscope (STM).¹⁵ With the performance characteristics described here, the operation of this STM with atomic resolution is possible seven days a week, 24 hours a day. Other instrumentation and activities that would benefit from the environmental control offered by such a laboratory might include high-resolution electron spectrometers, high-resolution electron microscopes, laser-based interferometers and vibrometers, atomically resolved scanning probe microscopes, advanced manufacturing machine tools, and the development of new, high-precision nanomanufacturing equipment.

For readers seeking additional information on the Hall Laboratory, Purdue University's Nanohub offers a web-based seminar (<http://www.nanohub.org/resources/3779/>) and a short video tour (<http://www.nanohub.org/resources/3795/>) describing the laboratory's layout, features, performance and characterization.

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ABOUT THE AUTHORS

As Facility Manager for the Birck Nanotechnology Center, **John R. Weaver II** oversees cleanroom and laboratory operations, facility infrastructure, training activities, and safety. Weaver received a BS in chemistry from Adrian College, and worked at RCA, Hughes Aircraft and Delphi Corporation before joining Purdue in 2006. He has published numerous papers in process development and contamination control, has two patents in process development, and authored a book and two book chapters on contamination control technology. He is a Senior Member of IEST, a recipient of IEST's Willis J. Whitfield Award, Chair of IEST Working Group 205: Nanotechnology Safety, and a member of the *Journal of the IEST* Editorial Board.

Mark Voorhis, Building Manager of the Birck Nanotechnology Center, is responsible for the overall operation of the building and its support systems. His principal goal is to make the operation of the facility transparent to the researchers, enabling them to achieve preeminence in their results. Voorhis has an AS in electrical engineering technology from Purdue, and 23 years of industrial experience in the construction, operation, and maintenance of cleanroom facilities. He has managed semiconductor process equipment and facility operations at the Round Rock, Texas, and Kokomo, Indiana, facilities of DuPont Photomask, Inc. He joined Purdue in 2004.

Ronald Reifenberger joined the Physics faculty at Purdue University in 1978 after completing post-doctoral studies at the University of Toronto. He obtained his MS and PhD in physics from the University of Chicago. Reifenberger has been active in research at the nanoscale since 1986. He has received the distinguished physics alumnus award from his alma mater (John Carroll University) in Cleveland, Ohio. Reifenberger's recent research interests include electron transport in nanostructures, vibrational properties of nanowires, and advanced applications of scanning probe microscopy. He has supervised more than 25 PhD students and has published over 180 papers in refereed journals.

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