NIST National Institute of Standards and Technology
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Standard Reference Material® 3461

Standard Reference Cantilevers for Atomic Force Microscopy Spring Constant Calibration

CERTIFICATE

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for validating methods for determining the stiffness of atomic force microscope (AFM) cantilevers as well as directly calibrating AFM test cantilevers using the reference cantilever method.

Description: A unit of SRM 3461 consists of one silicon microfabricated device approximately 1.6 mm by 3.0 mm containing an array of seven (7) uniform, rectangular cantilevers of varying length and stiffness at the end. Each cantilever is nominally 50 µm wide and 1.45 µm thick with lengths of 300 µm to 600 µm in steps of 50 µm (from left to right in the image in Figure 1, below, the lengths are 300 µm, 400 µm, 500 µm, 600 µm, 550 µm, 450 µm, 350 µm). Each unit has a unique serial number etched onto the lower portion of the device surface (see Figure 1 below). The device comes adhered to a polydimethylsiloxane (PDMS) gel film in a protective antistatic enclosure with a removable top. It can be used as-is by temporarily removing the top or could be carefully transferred to an appropriate alternative holder.

Certified Values: Certified stiffness values for each cantilever are provided below. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST [1]. These values are reported for each of the seven cantilevers on a single device which were independently measured. The certified values were assigned based upon measurements made at NIST using a laser doppler vibrometer (LDV) housed in a vibration, temperature, and humidity controlled clean room laboratory located 12 m underground. The apparatus has been modified for calibration and automation purposes.

The traceability of the LDV measurements to the International System of Units (SI) was verified through direct comparison of stiffness measurements of four random cantilevers using the NIST electrostatic force balance (EFB) in the Small Forces Group in the Physical Measurement Laboratory (PML), NIST, which is SI traceable. All values agreed to better than ±0.5 % which is a small contribution to the relative combined expanded uncertainty provided in this certificate which is between ±2.5 % and ±3.0 %. Details of the comparison of LDV to EFB stiffness measurements are provided in NIST SP 260-227.

Period of Validity: The certification of **SRM 3461** is valid indefinitely, within the measurement uncertainty specified. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified. Periodic recertification of this SRM is not required.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Table 1. Certified Values for SRM 3461
Device Sample

Nominal Cantilever Length (µm)	**Certified** Stiffness[a,b] (N/m)	**Certified** CE Stiffness Uncertainty[c] (N/m)	**Certified** Relative CE Stiffness Uncertainty[c] (%)
300	Sample	± Sample	± Sample
350	Sample	± Sample	± Sample
400	Sample	± Sample	± Sample
450	Sample	± Sample	± Sample
500	Sample	± Sample	± Sample
550	Sample	± Sample	± Sample
600	Sample	± Sample	± Sample

[a] The measured LDV stiffness values are certified and expressed as $x \pm U(x)$, where x is the value and $U(x)$ is the combined expanded (CE) uncertainty of the value. The true value of the measurand lies within the interval $x \pm U(x)$ with 95 % confidence. The value can be regarded as a normally distributed random variable with mean x and standard deviation $U(x)/2$.

[b] It should be noted that the stiffness values reported here are defined for the end of each cantilever. For uniform, rectangular cantilevers, stiffness increases cubically as the measurement loading point moves from the free end of the cantilever towards the base. The certified stiffness value of the reference cantilever must be divided by the off-end correction (OEC) factor at the measured (loading) point to provide the proper calibration stiffness:

$OEC_L = \left(1 - \frac{d}{L}\right)^3$, where d is the setback distance of the loading point from the end of the cantilever and L is the length of the cantilever.

[c] The combined expanded (CE) uncertainty values provided are based on the device population (n = 94) global pooled standard uncertainties of the LDV calibration measurements for each cantilever summed in quadrature with an estimated repeatability uncertainty and expanded using a coverage factor of $k = 2$. Details of this statistical analysis are provided in reference 3.

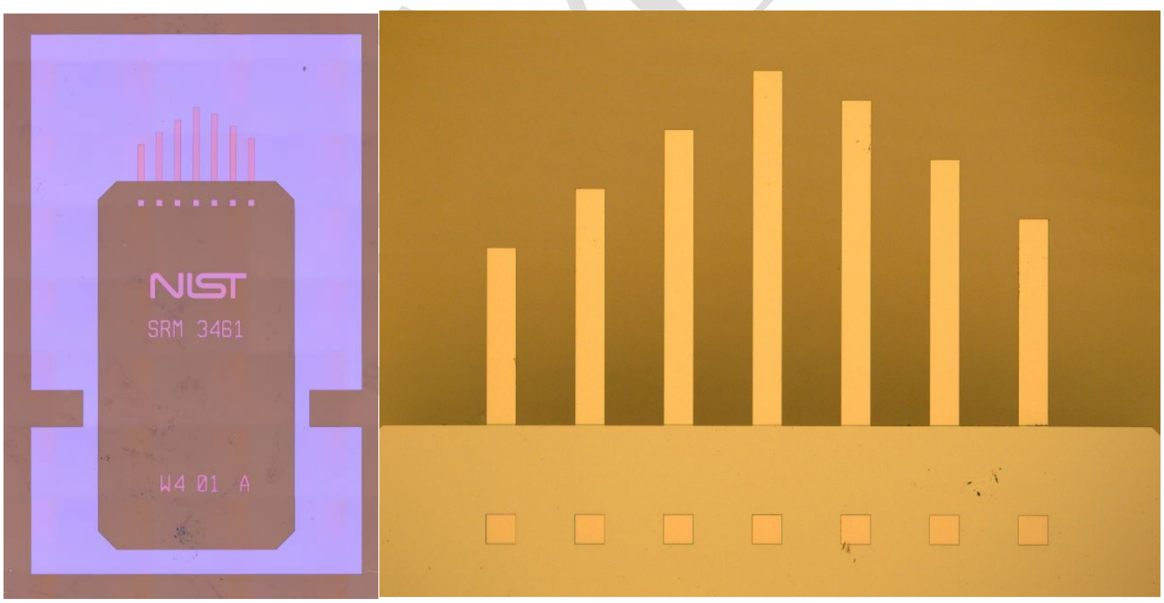

Figure 1. Example unit of SRM 3461.

Safety: SRM 3461 is made from single crystal silicon and is very inert. The cantilevers are small and relatively fragile and should not be touched except during carefully controlled calibration measurement processes. It is advisable to wear safety glasses when attempting to remount the device since improper handling with forceps may cause the chip to be flicked into the air, causing a potential eye hazard.

Storage: SRM 3461 was microfabricated, packaged, and calibrated in a clean room environment to minimize exposure to dust, debris, and contaminants. The packaging provided is intended to protect the device during shipping and storage in non-cleanroom laboratory environments. The device should be stored at room temperature (20 °C ± 5 °C) when not in use.

Use: The SRM 3461 device comes mounted on a PDMS gel film within a recessed anti-static polymeric tray with a removable clear cover. The device can be accessed by sliding the interlocked tray/cover unit out of the holder clip. The clear plastic top layer can then be pulled straight up, exposing the cantilever array within the tray. In some cases (e.g., LDV), the stiffness measurements can be conveniently made directly on each cantilever without removing the device from the tray. For use in calibrating test cantilevers in an AFM it is advisable to securely remount the device on an appropriate holder stub. This provides better access to a wider variety of AFMs as well as limiting movement of the chip itself when calibrating using the reference cantilever method where it is assumed that the cantilever flexes while the chip itself does not flex or move at all. Remounting and use of the AFM reference cantilever calibration method is described in more detail in reference 3, Appendix D.

Source and Preparation: SRM 3461 is microfabricated from a commercial 150 mm diameter silicon-on-insulator (SOI) wafer and designed to provide an extremely uniform series of cantilevers of differing stiffness as measured at the free end of each cantilever. The design and production of the devices from this wafer is described in detail in reference 3.

Analytical Approach for Determination of Cantilever Stiffness: Value assignment of the stiffness of each cantilever in SRM 3461 is based on a series of repeated measurements made by NIST using LDV and converted to stiffness using the Thermal Calibration Method [2]. This method is based on the equipartition theorem which equates the fundamental thermal energy of the first flexural mode resonance peak of the vibrational spectrum to the stiffness (potential energy) of the cantilever. Details of the LDV Thermal Calibration Method used to calibrate the stiffness of these reference cantilevers is provided in NIST SP 260-227.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Dec 2022).

[2] Gates, R.S.; Pratt, J.R.; *Accurate and Precise Calibration of AFM Cantilever Spring Constants Using Laser Doppler Vibrometry*; Nanotechnology, Vol. 23, p. 375702 (2012).

[3] Gates, R.S.; Osborn, W.A.; McLean, M.J.; Shaw, G.A.; Filliben, J.J.; *Certification of Standard Reference Material® 3461: Reference Cantilevers for AFM Spring Constant Calibration*. NIST Special Publication (SP) 260-227; U.S. Government Printing Office: Washington, DC (2022); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-227.pdf (accessed Dec 2022).

If you use this SRM in published work, please reference:

Gates RS, Osborn WA, McLean MJ, Shaw GA, and Filliben JJ (2022) Certification of Standard Reference Material® 3461: Reference Cantilevers for AFM Spring Constant Calibration. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-227. https://doi.org/10.6028/NIST.SP.260-227

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide the metrological traceability to the SI or other high-order reference system or have the required stability with time. These values are provided as "informational" values only. In the case of the first flexural resonance frequency values provided for each cantilever, while they can be both SI traceable and have LDV relative expanded measurement uncertainties lower than 0.1 %, they are susceptible to contamination during routine handling from debris and atmospheric particulates. While these contaminants do not change the stiffness of the cantilevers it may perturb the resonance frequency measurement which typically decreases when even tiny masses are added to the ends of cantilevers. The quality factor (Q) of the first flexural resonance frequency peak is also provided as an non-certified value and characterizes the sharpness of the peak which is inversely related to energy dissipation from the cantilever to the air around the cantilever. Q is the resonance frequency divided by the peak full width at half-maximum and is therefore dimensionless. Certified stiffness values for each cantilever are also repeated below.

Table A1. Certified and Non-Certified Values for SRM 3461
Device Sample

Nominal Cantilever Length (µm)	**Certified** Stiffness[a,b] (N/m)	**Certified** CE Stiffness Uncertainty[c] (N/m)	**Certified** Relative CE Stiffness Uncertainty[c] (%)	**Non-Certified** Resonance Frequency[d] (kHz)	**Non-Certified** Quality Factor[e] Q
300	Sample	± Sample	± Sample	Sample	Sample
350	Sample	± Sample	± Sample	Sample	Sample
400	Sample	± Sample	± Sample	Sample	Sample
450	Sample	± Sample	± Sample	Sample	Sample
500	Sample	± Sample	± Sample	Sample	Sample
550	Sample	± Sample	± Sample	Sample	Sample
600	Sample	± Sample	± Sample	Sample	Sample

[a] The measured LDV stiffness values are certified and expressed as $x \pm U(x)$, where x is the value and $U(x)$ is the combined expanded (CE) uncertainty of the value. The true value of the measurand lies within the interval $x \pm U(x)$ with 95 % confidence. The value can be regarded as a normally distributed random variable with mean x and standard deviation $U(x)/2$.

[b] It should be noted that the stiffness values reported here are defined for the end of each cantilever. For uniform, rectangular cantilevers, stiffness increases cubically as the measurement loading point moves from the free end of the cantilever towards the base. The certified stiffness value of the reference cantilever must be divided by the off-end correction (OEC) factor at the measured (loading) point to provide the proper calibration stiffness:

$OEC_L = \left(1 - \frac{d}{L}\right)^3$, where d is the setback distance of the loading point from the end of the cantilever and L is the length of the cantilever.

[c] The combined expanded (CE) uncertainty values provided are based on the device population (n = 94) global pooled standard uncertainties of the LDV calibration measurements for each cantilever summed in quadrature with an estimated repeatability uncertainty and expanded using a coverage factor of $k = 2$. Details of this statistical analysis are provided in NIST SP 260-227.

[d] Resonance frequency values were measured at NIST but are provided as informational only. They are non-certified but their relative expanded uncertainties are typically better than ±0.3 %. Resonance frequencies are susceptible to change from exposure to debris and other environmental particulate contaminants during use and handling.

[e] Quality factor (Q) values were measured at NIST but are provided as informational only. They are non-certified but their relative expanded uncertainties are typically better than ±5 %.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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