Resurrecting Dirty AFM Calibration Standards

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Abstract

Cleanliness of AFM samples before imaging has always posed questions for users. The same is true for height calibration standards. In this shop note, we discuss using CO_2 snow cleaning as a way to clean heavily contaminated standards and show its effectiveness in removing contamination. Further, we show that the cleaning does not damage the surface, nor alter the step heights, and can lead to improved imaging.

INTRODUCTION and BACKGROUND

Atomic Force Microscopy (AFM) analysts often face image distortions like streaks and unexpected height changes. Such imaging headaches are often related to contamination. CO_2 snow cleaning is a well known surface cleaning process which can remove contamination, and if applied here, it will lead to improved imaging. In this paper, we use the patterned surfaces of AFM calibration samples to show images of the same areas before and after cleaning. AFM step height calibration standards allow analysts to take raw data measurements and connect them to real world values. These standards provide both lateral and vertical calibrations and they are commonly used. However, these standards tend to get contaminated over time, making their usage and reliability subject to errors that can adversely affect any quantification.

These calibration standards come with few, if any, cleaning directions. Usually, manufacturers advise not to clean these samples, and AFM users have noted damage and stains from attempts to clean using solvents or ultrasonics. The purpose of this paper is to verify the effects of CO_2 snow cleaning by comparing AFM images made before and after the treatment on heavily contaminated AFM step height standards. Further, we demonstrate the calibrated step heights are unaltered by cleaning.

CO_2 snow cleaning is a well accepted surface cleaning process able to remove particulates of all sizes (down to 0.03 microns) and also hydrocarbons and organics[1]. Cleaning mechanisms, methodology, and examples have been described in the literature[2, 3, 4]. CO_2 snow cleaning of AFM samples has been discussed before and the removal of contamination was seen as a useful tool to improve images[5, 6]. However, no one has documented the cleaning of height standards with a focus on potential surface damage, alteration of calibration values, and image quality.

One important note regarding CO_2 snow cleaning needs clarification. CO_2 snow cleaning removes only items that are physically bound (via van der Waals or weak electrostatic forces) to the surface; any layer that is adherent to the surface should remain. Thin films deposited by evaporating, sputtering, or other methods where good adhesion is achieved should be undisturbed. Therefore, we expect the layers that define height standards to withstand the cleaning process. Contamination glued, seized, or buried into the surface will also remain.

EXPERIMENTAL PROCEDURES (MATERIALS AND METHODS)

We cleaned eleven samples and chose two for detailed AFM analyses:

- "18 nm step grid": VLSI Standards model STR10-180P surface topography reference. The pattern is a 2-dimensional square grid of pits with nominal pitch 10 µm and depth 18 nm. The material is silicon dioxide on silicon coated with a blanket thin film coating of platinum. The chip is secured to a steel disk using a conductive carbon sticky tab (double-side adhesive)[7].
- "180 nm step grid": Digital Instruments/Veeco Metrology "waffle" grid. The pattern is a 2-dimensional square grid of pits with nominal pitch 10 µm and depth 180 nm. The material is similar to the 18 nm step grid. The chip is secured to a steel disk using transfer adhesive.

Samples in the group of eleven ranged from 3 to 15 years old. Each had been used for checking AFM calibration. Most were used in an ordinary lab environment and stored in plastic boxes when not in use. One (specimen BN87A, a Digital Instruments 180 nm grid) was left uncovered in an ordinary office environment for more than 1 year. All of the specimens had defects or contamination when viewed in an optical microscope at 25-500x magnifications and some (such as BN87A) had debris visible to the unaided eye. At low magnification, the contamination pattern on BN87A resembled a fingerprint. At high magnification the contaminant gave distinct optical interference fringes and even hid portions of the pattern. Due to their condition, none of the specimens were currently being used for calibration. Therefore, all were candidates for this cleaning study.

A. The Atomic Force Microscope

The AFM system consisted of a Digital Instruments/Veeco Metrology NanoScope® IIIA controller, Electronics extender module ("phase box"), and Dimension 3100 large sample AFM fitted with a standard open-loop scanner. The Z calibration of the scanner is controlled using the working reference specimen in a traceable NanoDevices/VeecoProbes NanoGauge™ standard set. The working reference has a 202.16 nm step height. When comparing routine measurements over a period of weeks or months, we regard the uncertainty of the Z calibration as +- 2.5%. However, the short-term stability is much better and careful measurements made within a few hours show reproducibility better than 0.25% (0.5 nm at 200 nm). We used Olympus AC160 Silicon probes (300 kHz nominal resonant frequency). We operated the AFM in TappingMode™, capturing height and phase images of fields of view up to 15 µm. Note that the height data type presents ordinary topographic information and that the phase data type provides enhanced edge contrast and the possibility for material contrast.

Most of the AFM imaging was done at random spots. However, critical measurements of step height before and after cleaning were made on the same pit, verified by watching the position of the probe on the surface during scanning using the AFM's built-in video microscope.

B. Data Treatment

For display, images were usually flattened (by subtracting the line mean elevation and slope from each scan line) to eliminate low frequency noise and/or plane fitted to eliminate tilt. We measured step heights using the NanoScope offline step height analysis routine, which contains its own averaging and plane fitting methods. We measured bump height distributions using the grain analysis module of Image Metrology SPIP™ software ver. 4.7.1.

C. CO2 Snow Cleaning

CO_2 cleaning was performed using both the standard and high purity units from Applied Surface Technologies[8]. In the two sets of tests, we used either a liquid or a gas CO_2 source. In both cases, the

CO_2 purity was closer to industrial purity levels than to high purity. An asymmetric venturi-type nozzle was used for cleaning, which allows for a quasi-adiabatic expansion of the CO_2. This means we can use either a liquid or gas CO_2 source for cleaning. The advantages of a CO_2 gas source over a liquid source are that the stream is smaller, produces less snow and, subsequently, less moisture condensation and static charge. Generally, a CO_2 liquid source yields more snow and a larger cleaning area, giving a faster cleaning rate. This is useful for samples larger than typical calibration grids.

Sample disks were held with tweezers on a hot plate set at about 35-50 C. Cleaning distance from nozzle to sample was about 2 to 5 cm, and cleaning angles were both oblique and near perpendicular. Cleaning time was on the order of 1 to 5 seconds.

RESULTS AND DISCUSSION

Eleven samples were treated using CO_2 snow cleaning. All but one showed visual evidence of contamination removal based on examination under an optical microscope. The one that did not improve had previously been treated with cellulose acetate replication tape in a failed cleaning attempt. Five were imaged by AFM, and the contamination was almost completely removed in all cases. We will discuss three of these in detail.

A. Particle Removal

Figure 1 shows a tapping height and phase image of a random 15 μm field of view within the patterned area of sample BK88-A, an 18 nm step grid. The height image (left) shows a large number of small bumps distributed across the surface. The phase image (right) shows that these bumps have low phase relative to the surrounding substrate. Under our operating conditions, low phase indicates low stiffness and/or high adhesion. Based on this, we suggest that the bumps are a soft, organic contaminant. Figure 2 was captured 3 weeks after CO_2 snow cleaning. It shows another random 15 μm field. It is clear that the contaminating particles were almost totally removed. This result was typical for almost all of the contaminated calibration specimens.

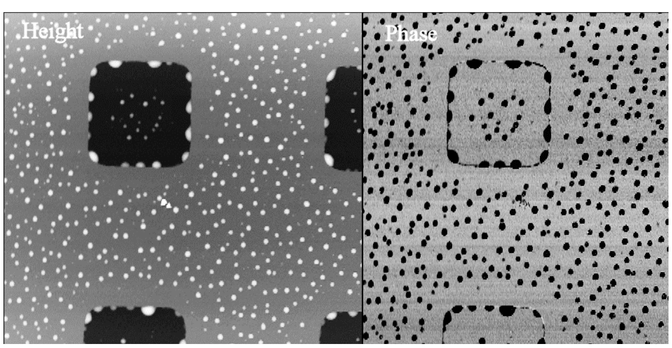

Figure 1. Height and Phase image of the contaminated surface of an 18 nm step grid. 15 μm scan.

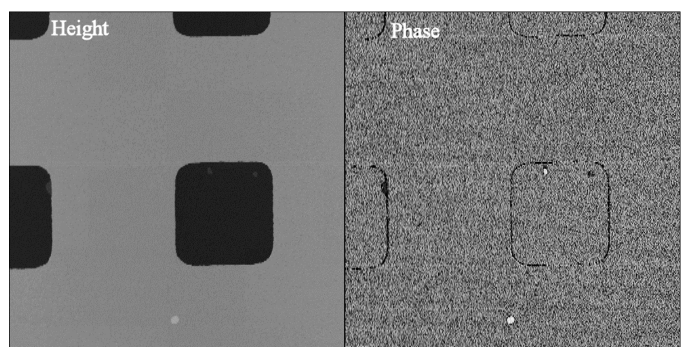

Figure 2. Height and Phase image, after cleaning. 15 μm scan.

Now consider the particle sizes. Referring to the “before cleaning” image (fig.1), we selected a 5.9x5.9 μm^2 area of land between 4 pits and measured the bump heights of all particles in that region which were taller than 3 nm above the mean substrate elevation. The 3 nm threshold was chosen to exclude surface roughness and noise spikes. The height distribution (fig. 3) was bimodal with the large particle peak at about 27 nm and the small particle peak below 5 nm. The particle density was $3.5/\mu m^2$. Fig. 4 shows a height profile crossing the lithographic step (measured here as 19.2 nm) and a particle almost twice as high (34.5 nm). Figure 5 shows a typical small particle was 4.8 nm high. After cleaning (fig. 2), we found only 4 particles in the entire 15x15 μm^2 field of view. The particle density was 0.018/μm^2, an apparent 99.5% reduction in overall particle density. All four particles were 4 to 5 nm high. Figure 6 shows a section plot across the step (measured here as 18.9 nm) and through one particle that was 4.6 nm high. For this sample, it appears that the treatment removed all particles taller than 5 nm and 96% of particles 3-5 nm high.

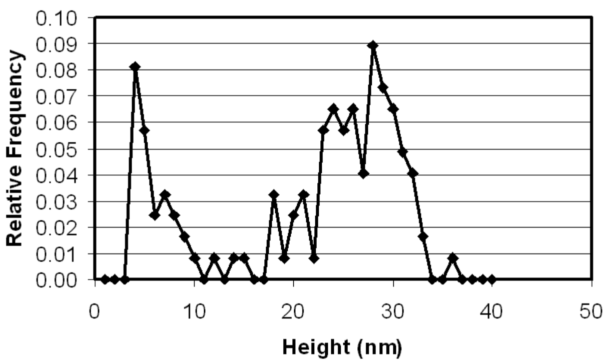

Figure 3. Histogram of bump heights on the 18 nm step grid before cleaning.

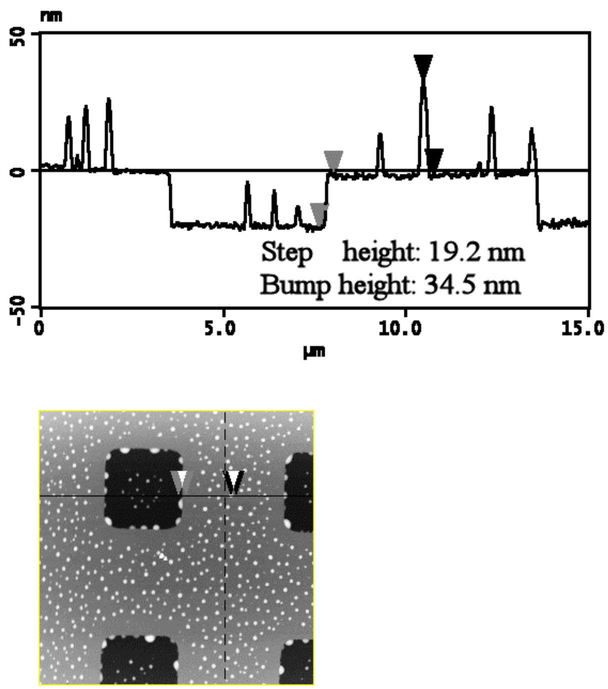

Figure 4. Height profile of the 18 nm step grid before cleaning, showing the measured step height (19.2 nm) and a typical tall particle almost twice as high (34.5 nm)

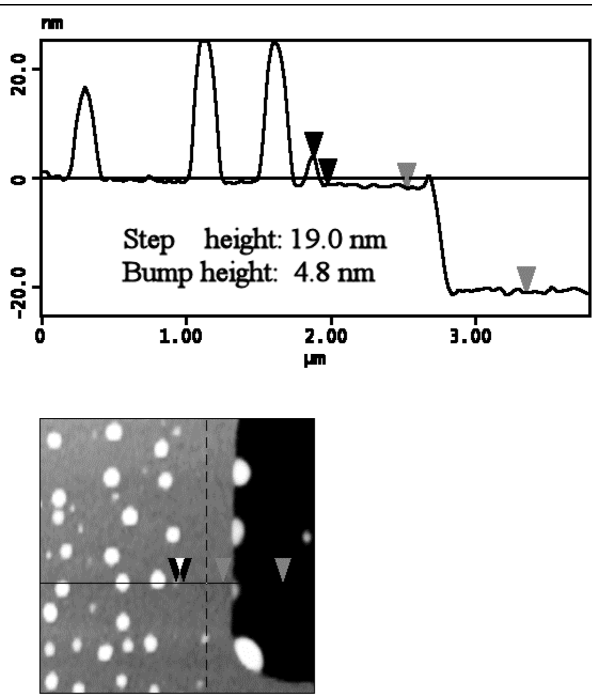

Figure 5. Another height profile before cleaning showing a typical small particle, 4.8 nm high.

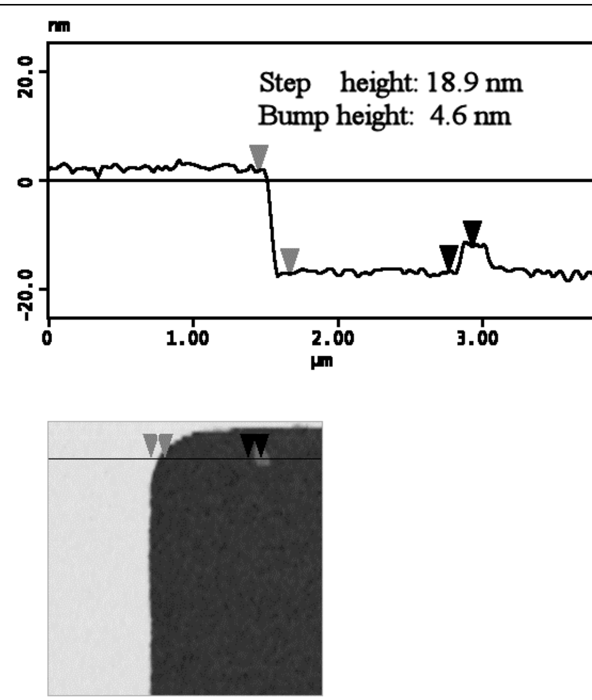

Figure 6. Height profile after cleaning showing a remaining particle, 4.6 nm high.

We note that cleaning did not remove all contamination, and some recontamination may have occurred because cleaning was done in ordinary, unfiltered air. Some of the samples we cleaned here may have had new particles but not enough to interfere with the conclusions or the analysis. Regardless of this residual or new contamination, specimens that had been quarantined were now almost as good as new.

B. Step Height Measurements.

It is reasonable to ask whether the cleaning treatment has any adverse effects on step heights. To answer this, we first measured step heights in clean areas on the contaminated and cleaned samples. For sample BK88-A (18 nm step), we found a step height of 19.13 nm before cleaning (fig. 7) and 19.58 nm after cleaning (fig. 8), a difference of 0.45 nm (2.17%). For sample AZ57-3 (180 nm step), we found a step height of 178.16 nm before cleaning (average of 2 pits) and 180.22 nm after (average of 4 pits), a difference of 2.06 nm (1.16%). Because these measurements were made months apart, they are subject to an uncertainty of +-2.5%. Although the before-after differences were less than this uncertainty, we decided to do a careful, same-day run of measurements to get a higher precision step height comparison. Using cleaned sample AZ57-3, we identified a specific pit and imaged the step height three times, withdrawing the probe and re-engaging between images. We then applied the CO_2 snow cleaning treatment and measured the same pit again, with three separate measurement cycles, as before. The averages of three measurements before and after were 180.803 and 180.707 nm, respectively, a difference of 0.097 nm (0.05%). All of these results show that the cleaning process does not alter step height.

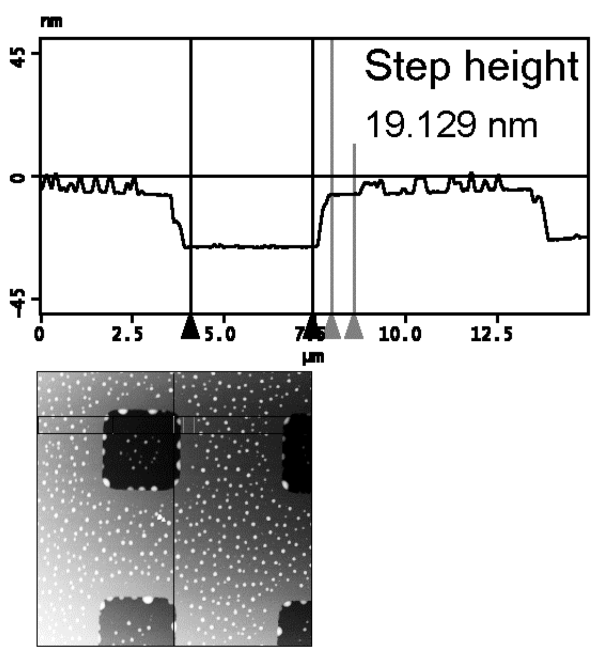

Figure 7. Step height measured before cleaning, using average lower and upper elevations in rectangular regions that were free of contaminant particles.

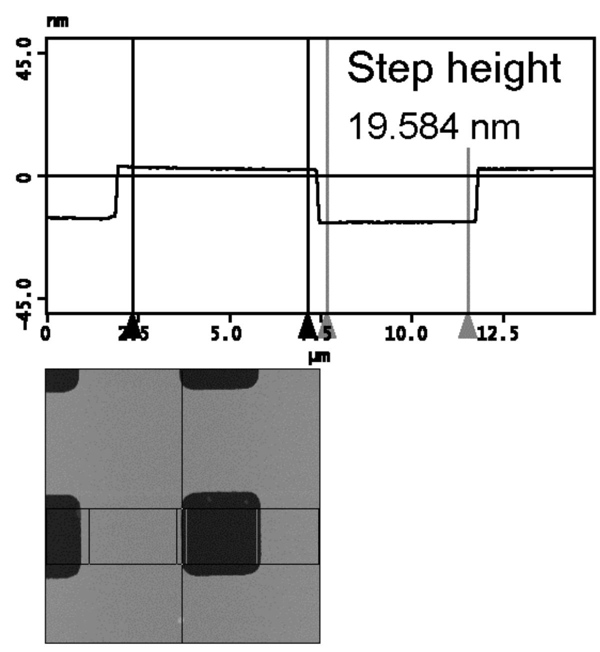

Figure 8. Step height after cleaning.

C. Imaging Improvements.

Bill Morris (6) of GE Corporate Research was perhaps the first to use CO_2 snow cleaning as an aid in cleaning AFM samples. He found that it removed particles and reduced the “nanoscum” on the surface. He said he got better images and fewer artifacts due to tip contamination. Previous demonstrations of imaging improvements before and after cleaning a given specimen were done using different spots. Here, to our knowledge, we show for the first time examination of the same spots on a single specimen. Fig. 9 shows images of a 2-μm pitch calibration pattern before and after CO_2 cleaning. Each image was scanned starting at the top of the frame and proceeding downward. The upper portion of panel A shows smooth flat areas and straight grooves, whereas the lower portion shows numerous horizontal streaks associated with vertical steps (different colors). Even after flattening (panel B), the image was still corrupted by horizontal streaks and shifts in the groove edges. Horizontal and vertical shifts are due to material transfer to and from the probe tip during the scan. After replacing the contaminated probe and cleaning the specimen, we captured the height image shown in panel C. This image shows smooth flat areas and straight grooves throughout, without flattening.

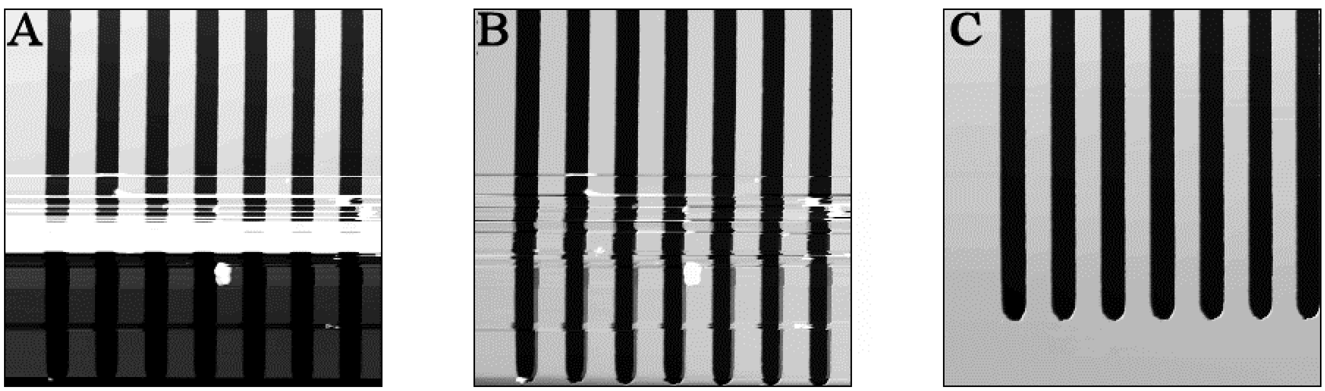

Figure 9. Height images of the same area of a 200 nm step grid before and after cleaning. A: Height image before cleaning, as captured. B: Flattened height image. C: Height image after cleaning, as captured. 15 μm scans.

Lest the reader be tempted to use CO_2 snow cleaning on every AFM specimen, we urge some caution. This cleaning may be acceptable in many applications. But when the surface features cannot survive cleaning or the contamination is itself of interest then one should not clean.

CONCLUSIONS

In this work, we applied CO_2 snow cleaning to a panel of AFM calibration samples that have distinct topographic patterns and calibrated step heights. We showed that both visible and even nanoparticle contaminants, which are commonly found on aged specimens, can be removed. Next, we showed that the structure was preserved so that the step height remained the same. Finally, we demonstrated the imaging improvements (reduction of image artifacts and tip destruction) that make it rewarding to use this cleaning treatment. In summary, we have shown that CO2 snow cleaning can revive and refurbish dirty calibration grids without any structural damage so that they can be used again to provide consistent and reproducible quantitative calibration data.

REFERENCES

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(6) Bill Morris, private communication, see www.co2clean.com/afm.htm

(7) vlsistd.com/products/dimensional/streferences.asp?sid=31

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Pacs numbers: 07.790Lh, 82.37.GK, 81.65CF