DIMENSIONAL MEASUREMENTS IN THE NANOMETRIC RANGE

P. Herbert Osanna
Laboratory for Nanotechnology
Department for Interchangeable Manufacturing and Industrial Metrology - AuM
Vienna University of Technology, A-1040 Wien, Austria
Phone: +43 1 58801 31140 Fax: +43 1 58801 31196 E-mail: osanna@mail.ift.tuwien.ac.at

Abstract
Since about 1982 new high resolution and high precision measurement methods have been developed whereas these methods can be used especially for the evaluation of surface topography in the nanometric range. Results of a series of experiments are analysed and discussed. General conclusions can be drawn as very often obtained scanning images looking like “atoms” topography structure have been misunderstood, because it is very difficult to interpret correctly and properly so many kinds of SPM artefacts in the imaging practice applications.

Key Words: Micro and nano technology, nano metrology, image topography, SPM artefacts

1. Introduction

Increases of the quality of products are not to be joined of course exclusively with increase of accuracy but up to a certain extent correlation is given, particularly if we look on the technical development during the last century [1, 2]. This trend develops presently continuously further on because of the general development from micro technology to "nano technology" under which particularly special measurement techniques and production methods are to be understood for the realisation of manufacturing accuracy in the nanometric range [3, 4].

It is emphasised, that in this respect applications in micro electronics do not stand in the focal point. Rather instruments of mechanical engineering and particularly precision engineering are addressed in the first hand. Extremely high accuracy demands deposit presently already at highly developed instruments for everyday use as there are VCRs or CD-players and in the sensor technique in automotive engineering and even in the home appliance if we think on one-hand mixing taps which demand ultra precision form tolerances.

In persecution of this aim since about 1982 new high resolution and high precision measuring devices have been developed, especially Scanning Tunnelling Microscopy (STM) [5] and Atomic Force or Scanning Probe Microscopy (AFM, SPM). For highest demands these methods make it possible to explore atomic structures and in general very accurate and small industrially produced parts and structures [6]. Figure 1 shows a 3-D image topography of a sample surface of a copper alloy "atomic“ structure.

2. Evaluation of the Topography of Ultra-Precision Surfaces

Currently, it is known that the evaluation of the topography of ultra-precision surfaces can be carried out by using Scanning Probe Microscopes (SPM). Scanning Probe Microscopy is a powerful technique, offering the ability to gain surface topography information that is not available in any other way, and relying on a mechanical probe for the generation of magnified images [7, 8].

The ideal microscope will generate a direct image map of the sample surface, such that the measurements which are made will be an exact magnification of the actual sample. Because of magnifying in three dimensions, the ideal SPM will provide measurements in all dimensions that are accurate representations of the sample surface.

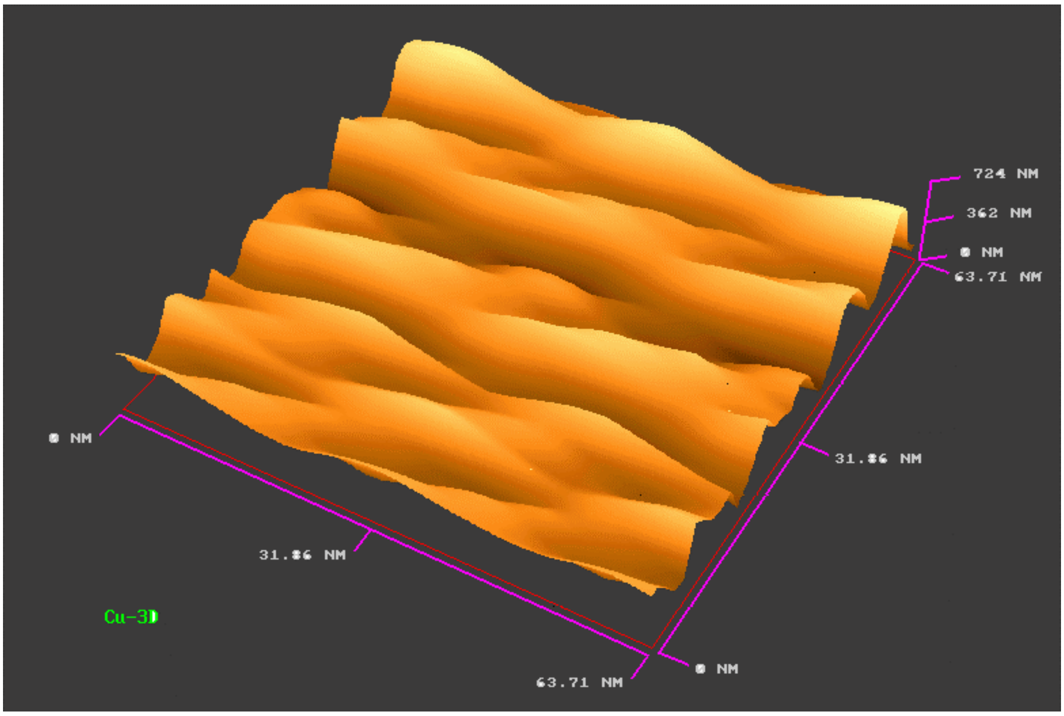

Figure 1: 3-D Image Topography of a Sample Surface of a Copper Alloy Atomic Structure

All analytical techniques contain artefacts [9]. The artefacts of traditional instrument techniques become better understood and are minimised by changes to the instrumentation, methodology or by knowledgeable interpretation of data. SPM is also subject to artefacts. If these are not understood, the SPM data can not be properly interpreted, led to many undesirable consequences, such as the inability to properly evaluate instrument performance and improper use of analytical results. The artefacts in SPM are not easy to be understood, because of resulting from very complicated sources and the sensitivity of the image results.
It is also very important that the operation performance, operator experiments and limitations of SPM instrumentation should be understood in order to properly interpret the images obtained. When artefacts are well understood, SPM data can be properly interpreted and the information can be used with confidence.
The confidence in SPM imaging results with influence of the artefacts and calibration methods of SPM instrumentation is always barrier to realise wide industrial application of SPM technology.

3. Topographies with Nanometric Resolution

AFM (Atom Force Microscope) is one kind of SPM technique. AFM images are a combination of the sample surface and the probe that is used to image the micro and nano surfaces. An AFM is operable in ambient air, liquid or vacuum to solve features in three dimensions down to a fraction of an angstrom and is composed of a sensing probe, piezoelectric ceramics, a feedback electronic circuit, and a computer for generating and presenting images.
The measurement principle of AFM is, that AFM uses the force sensor to measure the deflection of a cantilever. The cantilever composed of a narrow (120 or 200 µm) silicon nitride arm with integrated tip scans the surface of the sample in form of grid-pattern. The scanning process is controlled by piezoelectric ceramics (x, y) with a resolution of 0.1 angstrom. The laser beam is focused on the upper side of the cantilever through a optical system and then reflected to a four section photo detector. The amount of the motion of the cantilever can then be calculated from the difference in light intensity on

the sectors. The signal of the cantilever motion will be led into the feedback loop, which controls the third movement of the piezo actuator in z-direction. The changes of z-position of the cantilever, which reflect the surface structure of the sample, are taken as the measuring signals and then processed for generating the image topographies.

Hooke's law gives the relationship between the cantilever motion z, and the force required to generate the motion, F:

$$F = - K.x \qquad (1)$$

K is the force constant of the cantilever, and taken as 1 Newton/meter.

The image topographies of the ultra precisely milled surfaces of Al- and Cu-alloys generated by using a diamond tool on an air-bearing precise milling machine as well as silicon coated with C-N ion-beams were obtained and analysed by means of AFM.
The sample materials are OFHC-copper and AlMgSi 13. The dimensions are: diameter = 48 mm, height = 8 mm; the surface roughness values at the cross-sectional areas are: Ra < 10nm, Rmax < 100nm.
Figures 2 shows the typical image topography and the analytical image results of one of the test surfaces.

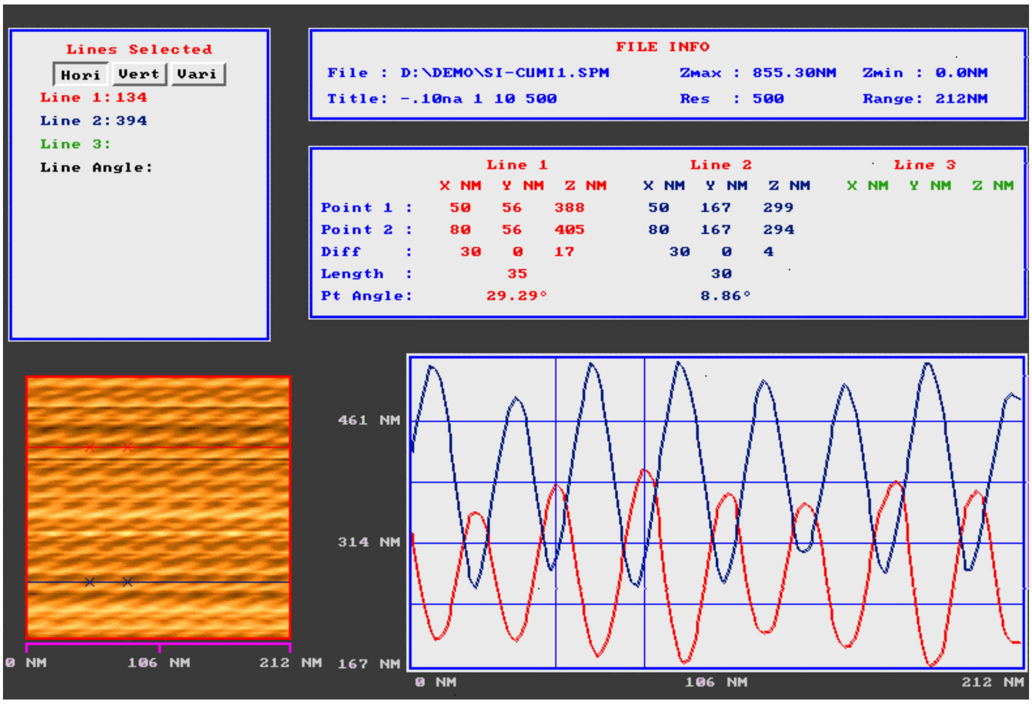

Figure 2: Surface image results of copper alloy

4. Artefacts

Any measurement that results in an image differing from the actual sample surface is an artefact [9, 10]. Artefacts are a part of any microscope technique. SPM artefacts can come from many sources. These artefacts are ranged over probe artefacts, scanner and piezoelectric ceramic artefacts, artefacts

resulting from instrument mechanical and electronic design, artefacts resulting from data manipulation and artefacts created by SPM samples.
As mentioned above, artefacts in SPM images can result from probe, such as probe/cantilever artefacts, probe geometry, step/concave feature distortions, convex feature distortions, probe asymmetry, probe flexing, cantilever backlash, probe reproducibility, contamination, double tips, and probe/sample angle.
The scanner and piezoelectric ceramic artefacts can come from the scanner material characters and the mechanical construction of SPM scanners, such as non-linearity, drift, creep, hysteresis, bowing, and dynamic range artefacts of the piezoelectric ceramics.
Artefacts resulting from instrument mechanical and electronic design are from for example, vibration between the SPM probe and sample, thermal stability of the stage, optimisation of the feedback control loop, instrument noise from the SPM power supply. Vibration resulting from instrument mechanical and electronic design is also a main factor resulting in SPM artefacts. When vibration occurs between the sample and the SPM probe, they can produce difficulty in achieving stable feedback and artefacts in images. Images which appear to look like ”atoms“ have actually been due solely to vibration [9]. These artefacts can appear as oscillations at the vibration frequency.
Data manipulation artefacts come from enhancement techniques of SPM images, such as tilt and level correction, band pass filtering, fast Fourier Transform, smoothing algorithms, shading and independent x, y, and z magnification.
Artefacts created by SPM samples are from, such as contamination of the sample, sample curvature, softness of the sample, probe/sample adhesion, particles on the sample surface and features deeper than the probe length.
The scanning process of nano surfaces by using AFM is also often influenced by several kinds of external factors, especially by environmental influences like vibrations, sound, air flow, changes of temperature, etc. They appear also as distortion of images or errors of measurements of the microstructures.

5. Analysis of Affect Factors of Nano Scanning Images

The imaging results of a group of experiments that deal with imaging two ultra precise milled samples composed of Cu- and Al-alloys, and Silicon coated with C-N ion-beams in 1µm scan range, using scanner which has a maximum scan range of 150µm on AFM that has been installed on an anti-vibration worktable in a precise measuring laboratory, in which the temperature is maintained constant 20±0.1°C, have been achieved and will be discussed as following. The base of the Laboratory was also made for anti-vibration. The scanning head and scanning stage of the AFM are masked in a glass-plastic mask without the influence of air flow. Therefore, it is desirable to image micro and nano structures of ultra precisely machined surfaces by using AFM. The influence from the external factors in this case can be ignored.
For the all samples, the same experiment processes were taken, and the scanning range is always 1□m, and other scan parameters are also same.
By means of the analysis of the image results, an interesting phenomenon that the image topographies obtained from different samples with the scanning parameters selected in a certain area are just like the same one - “atoms” structure, being independent of the surface states, manufacturing techniques and materials of the samples, appears. Images which appear to look like ”atoms“ have actually been due to the artefacts mentioned above in chapter 4, especially due to the insufficient high resolution of scanner and the control system of the scanner, and mainly due to vibration resulting from mechanical and electronic design of the instrument, e.g. the vibration of the scanning movement between the scanner, the tip of the probe and the sample, because these artefacts in SPM images can appear as oscillations at the vibration frequency through analysis of the image topographies obtained. This can be determined by varying the scan speed, range and rotation, and observing the resulting change in the image. The frequency of the vibration has been obtained through the investigation. It is about 350 Hz.
The influence grade of the vibration is dependent of the scanning frequency and size of the scanning area. If the vibration between the probe and sample appears, it is very difficult to get the stable

feedback and the artefacts also appear in the image results. The operator experiences are also very important for the adjustment of the scanning parameters at nanometric resolution.

Scanning images of the nano surfaces using probe on AFM is often influenced with several kinds of causes as mentioned in chapter 4. There exist environmental influences like ambient vibrations, sound, air flow, changes of temperature etc. but also other influences as there are e.g. noise from sensor (scanner) devices, geometry of scanning probes, and scanning movement itself. Measurement errors due to scanning movement are caused by non-linearity and hysteresis of commonly used piezoelectric-translators. They appear as distortion of images or errors of measurements of the microstructures.

If the feedback control loop is not properly optimised, oscillations or other undesired effects may result. These can cause the SPM image to show waves, noise, or other artefacts. Noise from the SPM power supply will cause noise on the voltages applied to the piezoelectric ceramics.

The techniques that are used to enhance SPM images may also distort the data and lead to misinterpretation. Tilt and level correction, band pass filtering; Fast Fourier Transform (FFT), smoothing algorithms and shading, which can be helpful in enhancing SPM images, can also introduce artefacts.

Sample contamination and curvature can also result in artefacts, particularly if combined with sample tilt. Softness, probe/sample adhesion, particles on the sample surface and features deeper than the probe length can create the artefacts. If a feature is imaged, such as a pit, which is deeper than the probe tip is long, the cantilever will strike the edge of the feature. The resulting image will be a combination of the actual sample surface and the shape of the cantilever.

An understanding of the interaction between probes and samples is important, in order to properly evaluate SPM images. Obtaining reproducible tips is currently a major challenge, especially since good tips often become dull or dirty after 1 day or less of imaging.

The scan range of considerable software manipulation is possible, making poor raw data appear to be of high quality. This does not mean that the data are an accurate representation of the sample surface, however, and must be interpreted with caution. A knowledge of the image rendering techniques used and access to the raw data collected are essential for analyst to interpret SPM data with confidence and present quality images.

The artefacts and errors can be reduced in several ways: by tubular corrections or, by used of position sensors and interpolation between nominal values and actual sizes or, by a priori high precision positioning, that is positioning of piezo-translators in a closed loop control. Use of closed loop controlled piezo-translators allows to reduce non-linearity, depending on performance of positioning sensors and control circuits This requires the compromise between decreasing scanning speed and increasing positioning accuracy. In order to obtain the image topographies with atomic resolution by using AFM, the artefacts mentioned in chapter 3 must be avoided and properly interpreted.

6. Discussion

In this paper, the sources of SPM artefacts and the affect factors of the SPM images are presented and investigated. The imaging results obtained have been analysed and discussed, and the frequency of the vibration from sensor devices and scanning movement are investigated..

It has been concluded that so many scanning images looking like “atoms” topography structure obtained have been misunderstood in the world, because it is very difficult to correctly and properly interpret so many kinds of SPM artefacts in the imaging practice applications. Therefore, it is very important to control the vibration between the sample and the SPM probe, e.g. to improve the design and manufacturing technology of new types of scanners and nano instrumentations to enhance the wide industrial application of SPM technology.

For the future development of the SPM instrumentation, the characters of the piezoelectric ceramics, such as hysteresis, creep and thermal drift must be perfectly avoided or compensated, and the all artefacts mentioned above and the influences of the environment, e.g. the influence of the temperature, the air flow and the vibration have to be minimised and be understood, the images obtained must be properly interpreted.

The confidence in SPM imaging results with influence of the artefacts and calibration methods of SPM instrumentation are always barriers to realise wide industrial application of SPM technology.
In order to obtain the image topographies with atom resolution by using AFM, the resolution and the stability of the piezoelectric ceramics, the vibration of the scanning movement between the probe and sample and the geometry of the probe as well as the operator experiences play a very important role.

7. Conclusions and Final Remarks

Today the scanning probe methodology is still partially under developing phase of the measuring technology. It is sure that AFM is a powerful instrumentation for nano surface measurement in the future. But the confidence in SPM images, limitation of the calibration method for AFM and the stability of the scanning results are always the barrier to realise the fully industrial applications of SPM technology.
In any case the developments in nano metrology and instrumentation are fully underway. They will continue in the future continually further on and a possible end is to be foreseen in no way. On the contrary in continuation of the already by Kienzle and Taniguchi demonstrated trend the development will continuously lead from "Nano Technology" to "Pico Technology".

References:

[1] Kienzle, O.: Genauigkeitsansprueche des Konstrukteurs und ihre Verwirklichung durch die Fertigung. Industrieanzeiger 82 (1960), No 62, pp 26/42
[2] Osanna, P. H.: Dreidimensionales Messen. Future 80, Frankfurt: Ingenieur-Digest-Verlag, pp 216/218
[3] Taniguchi, N.: On the Basic Concept of Nanotechnology. Proc. Int. Conf. Prod. Eng. Tokyo, 1974, part 2, pp 18/23, Tokyo: JSPE.
[4] Whitehouse, D. J.: Nanotechnology Instrumentation. Measurement + Control 24 (1991), No.2, pp 37/46
[5] Binnig, H., Rohrer, H.: Scanning Tunnelling Microscopy. Helv. Phys. Acta 55 (1982), pp 726/731
[6] Si, L., P.H. Osanna, N.M. Durakbasa, D. Prostrednik: Die Untersuchung der Mikrostruktur von Kompakt-Disks durch Anwendung von AFM. Proceedings of 5th International DAAAM Symposium, Maribor, SLO, 1994, pp 405/406
[7] Y. Ichida, K. Kishi: Nanotopography of Ultraprecise Ground Surface of Fine Ceramics Using Atomic Force Microscope. *Annals of the CIRP*, 42/1 (1993), pp 647/650.
[8] L. Si, P. H. Osanna: Nano-Oberflaechen-Fertigungsverfahren und -Untersuchungsmethoden. In: P. H. Osanna and M. Skopal (Editors): Nanotechnologie - Nanometrologie, TU Brno, CZ, 1995, p 43/53.
[9] P. H. Osanna, L. Si, D. Prostrednik: Problemanalyse der Atomkraftmikroskopie-Aufnahmen unter dem Blickpunkt der Praezisionsmesstechnik. Proceedings of BIAT'96, Zagreb, Croatia,1996.
[10] User´s Manual of TMX 2000 Scanning Probe Microscope. Version 3.05, TopoMetrix, Santa Clara, USA.