AFM AS AN ADVANCED TECHNOLOGY IN DIAGNOSTICS OF METALLIC MATERIAL PROPERTIES

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Abstract

This article deals with the assessment of the atomic force microscopy (AFM) advantages in the diagnosis of metallic materials properties. The advantages of AFM are compared with traditional metallographic procedures and also with the possibilities of scanning electron microscopy (SEM). There are compared the results of measurements on samples of alpha brass CuZn30 in this article. Until at metallographic technique we can obtain information about the macrostructure on microns level, SEM is doing structural microanalysis at the nanometer level and using the AFM brings us possibility to study materials at the atomic level.

Keywords: Atomic Force Microscopy (AFM), material diagnostics, Scanning Electron Microscopy (SEM), nanostructural properties of metallic materials

1 Introduction

Atomic force microscopy is arguably the most versatile and powerful microscopy technology for studying samples at nanoscale. It is versatile because an atomic force microscope can not only image in three-dimensional topography, but it also provides various types of surface measurements to the needs of scientists and engineers. It is powerful because an AFM can generate images at atomic resolution with angstrom scale resolution height information, with minimum sample preparation.

2 Atomic force microscopy

For imaging, the reaction of the probe to the forces that the sample imposes on it can be used to form an image of the three-dimensional shape (topography) of a sample surface at a high resolution. This is achieved by raster scanning the position of the sample with respect to the tip and recording the height of the probe that corresponds to a constant probe-sample interaction (see section topographic imaging in AFM for more details). The surface topography is commonly displayed as pseudocolor plot. An AFM image is a simulated image based on the height of each point of the surface and, in fact, each point (x, y) of the surface has a height h(x, y) [1].

Applications in the field of solid state physics include the identification of atoms at a surface, the evaluation of interactions between a specific atom and its neighboring atoms, and the study of changes in physical properties arising from changes in an atomic arrangement through atomic manipulation.

2.1 Configuration

An AFM typically consists of the following features (Fig.1). The small spring-like cantilever (1) is carried by the support (2). Optionally, a piezoelectric element (3) oscillates the cantilever (1). The sharp tip (4) is fixed to the free end of the cantilever (1). The detector (5) records the deflection and motion of the cantilever (1). The sample (6) is mounted on the sample stage (8). An xyz drive (7) permits to displace the sample (6) and the sample stage (8) in x, y, and z directions with respect to the tip apex (4). Although Fig.1 shows the drive attached to the sample, the drive can also be attached to the tip, or independent drives can be attached to both, since it is the relative displacement of the sample and tip that needs to be controlled. Controllers and plotter are not shown in this figure. Coordinate directions are defined by the coordinate system (0) [2].

A cantilever (1) with a sharp tip (probe) at its end is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. On the Fig.2 and the Fig.3 there are shown details of an AFM cantilever.

When the tip of the cantilever is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law [3]. Depending on the situation, forces that are measured in AFM include mechanical contact force, van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces, solvation forces, etc.

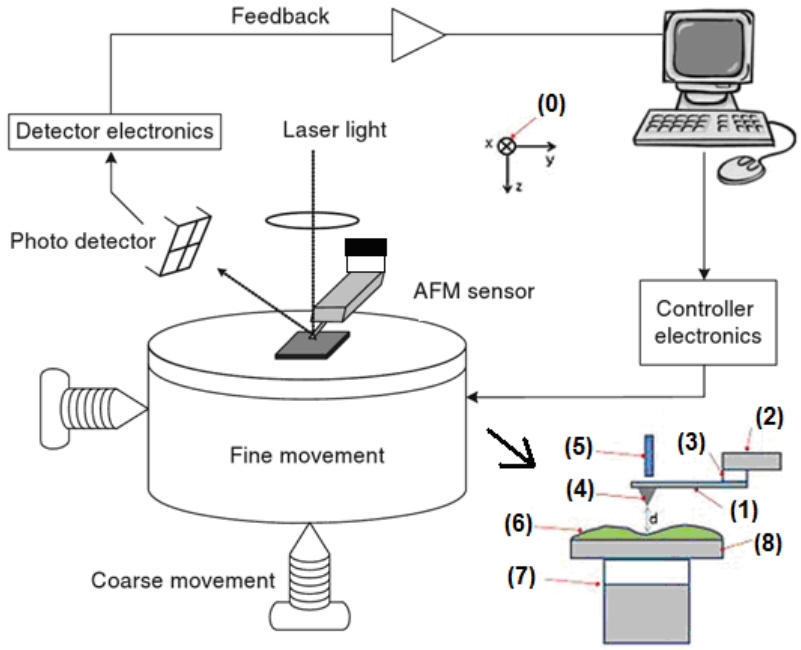

Fig. 1 Typical configuration of an AFM [4]

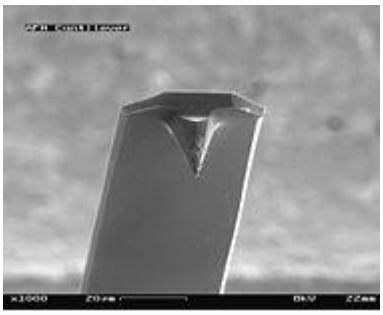

Fig. 2 *Electron micrograph of a used AFM cantilever. Image width ~100 micrometers*

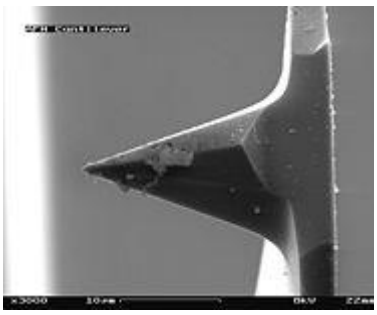

Fig. 3 *Electron micrograph of a used AFM cantilever. Image width ~30 micrometers*

According to the configuration described above, the interaction between tip and sample, which can be an atomic scale phenomenon, is transduced into changes of the motion of cantilever which is a macro scale phenomenon. Several different aspects of the cantilever motion can be used to quantify the interaction between the tip and sample, most commonly the value of the deflection, the amplitude of an imposed oscillation of the cantilever, or the shift in resonance frequency of the cantilever.

The AFM can be operated in a number of modes, depending on the application. In general, possible imaging modes are divided into static (also called *contact*) modes and a variety of dynamic (non-contact or "tapping") modes where the cantilever is vibrating or oscillating at a given frequency [5]. There are two dynamic modes: tapping mode, also called intermittent contact, AC mode or vibrating mode (amplitude modulation AFM) and non-contact mode (frequency modulation AFM). It should be noted that despite the nomenclature, repulsive contact can occur or be avoided both in amplitude modulation AFM and frequency modulation AFM, depending on the settings.

2.2 Contact mode

In contact mode, the tip is "dragged" across the surface of the sample and the contours of the surface are measured either using the deflection of the cantilever directly or, more commonly, using the feedback signal required to keep the cantilever at a constant position. Because the measurement of a static signal is prone to noise and drift, low stiffness cantilevers (i.e. cantilevers with a low spring constant, k) are used to achieve a large enough deflection signal while keeping the interaction force low. Close to the surface of the sample, attractive forces can be quite strong, causing the tip to "snap-in" to the surface. Thus, contact mode AFM is almost always done at a depth where the overall force is repulsive, that is, in firm "contact" with the solid surface.

Working in contact mode, the tip is brought into contact with the sample, and the sample is raster scanned along an x-y grid (Fig. 1). Most commonly, an electronic feedback loop is employed to keep the probe-sample force constant during scanning. This feedback loop has the cantilever deflection as input, and its output controls the distance along the z axis between the probe support (2 in Fig. 1) and the sample support (8 in Fig. 1). As long as the tip remains in contact with the sample, and the sample is scanned in the x-y plane, height variations in the sample will change the deflection of the cantilever. The feedback then adjusts the height of the probe support so that the deflection is restored to a user-defined value (the set point). A properly adjusted feedback loop adjusts the support-sample separation continuously during the scanning motion, such that the deflection remains

approximately constant. In this situation, the feedback output equals the sample surface topography to within a small error.

The AFM signals, such as sample height or cantilever deflection, are recorded on a computer during the x-y scan. They are plotted in pseudocolour image, in which each pixel represents an x-y position on the sample, and the color represents the recorded signal.

2.3 Taping mode

In ambient conditions, most samples develop a liquid meniscus layer. Because of this, keeping the probe tip close enough to the sample for short-range forces to become detectable while preventing the tip from sticking to the surface presents a major problem for non-contact dynamic mode in ambient conditions. Tapping mode was developed to bypass this problem [6]. Nowadays, tapping mode is the most frequently used AFM mode when operating in ambient conditions.

In tapping mode, the cantilever is driven to oscillate up and down at or near its resonance frequency. This oscillation is commonly achieved with a small piezo element in the cantilever holder, but other possibilities include an AC magnetic field (with magnetic cantilevers), piezoelectric cantilevers, or periodic heating with a modulated laser beam. The amplitude of this oscillation usually varies from several nm to 200 nm. In tapping mode, the frequency and amplitude of the driving signal are kept constant, leading to a constant amplitude of the cantilever oscillation as long as there is no drift or interaction with the surface. The interaction of forces acting on the cantilever when the tip comes close to the surface, Van der Waals forces, dipole-dipole interactions, electrostatic forces, etc. cause the amplitude of the cantilever's oscillation to change (usually decrease) as the tip gets closer to the sample. This amplitude is used as the parameter that goes into the electronic servo that controls the height of the cantilever above the sample. The servo adjusts the height to maintain a set cantilever oscillation amplitude as the cantilever is scanned over the sample. A tapping AFM image is therefore produced by imaging the force of the intermittent contacts of the tip with the sample surface [7].

Although the peak forces applied during the contacting part of the oscillation can be much higher than typically used in contact mode, tapping mode generally lessens the damage done to the surface and the tip compared to the amount done in contact mode. This can be explained by the short duration of the applied force, and because the lateral forces between tip and sample are significantly lower in tapping mode over contact mode.

When operating in tapping mode, the phase of the cantilever's oscillation with respect to the driving signal can be recorded as well. This signal channel contains information about the energy dissipated by the cantilever in each oscillation cycle. Samples that contain regions of varying stiffness or with different adhesion properties can give a contrast in this channel that is not visible in the topographic image. Extracting the sample's material properties in a quantitative manner from phase images, however, is often not feasible.

3 Experimental results

In our experiments, we examined samples of α-brass CuZn30 with chemical composition according to DIN17660.W, No. 2.0256. The chemical composition of this alloy is presented in Table 1. The volume of all impurities is less than 0.5 wt.%. From the structural point of view, a single-phase structure of this alloy is composed as a solid solution α. Cu crystallizes in face centered cubic (FCC) structure. Zn crystallizes in hexagonal close-packed (HCP) structure. The lattice constant of Cu is a = 0.36146 nm and Zn lattice is a = 0.2665 nm. The lattice constant of alloys is usually changed due to arrangement of individual atoms in the crystal lattice.

Table 1 *The chemical composition of the studied alloy CuZn30 (Ms70) by DIN 17 660*

Alloy wt. %]	Cu	Al	Fe	Ni	Pb	Sn	other	Zn
CuZn30	69.0 – 71.0	0.02	0.05	0.2	0.05	0.05	0.1	rest

3.1 Preparing of the samples

The samples presented in the experimental section were grinded on a diamonds disks down to 2 500 grit using a lubricating emulsion then were polished. Diamond suspensions with 9 μm, 3 μm and 1 μm particle size on a cloth and the suspension colloidal silica 0,05 μm particle size were used for polishing. After polishing the samples were etched.

3.2. Results from optical microscope and SEM

To compare the advantages of the AFM, the surface topography of the samples was monitored by optical microscopy NEOPHOT 32 and scanning electron microscopy (SEM). We have used the high resolution scanning

electron microscope JEOL 7600F boasting all the analytical modules for detailed chemical and microstructure analysis of metal alloys.

The results of monitoring are shown in Fig. 4 to 7. Fig. 4 shows a sample topography obtained by the optical microscope NEOPHOT 32 at optical magnification of 500. We can clearly see the individual crystalline grain structure and their mutual relations. Here we can follow the structural defects in sizes comparable to the size of the crystal grains. The comparable pictures viewed from the SEM are achieved using the lower detector of secondary electrons (SE) (Fig. 5). Details of the structure are less traceable using the upper SE detector, as it is seen in Fig. 6. The higher magnification of SEM leads in a loss of information about the topography of the sample surface (see Fig. 7).

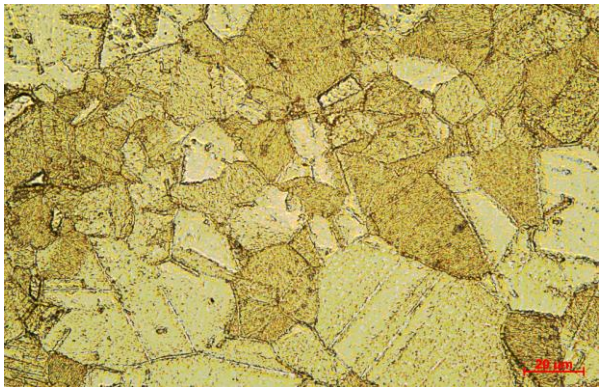

Fig. 4 *Topography of brass CuZn30 surface from optical microscope NEOPHOT 32*

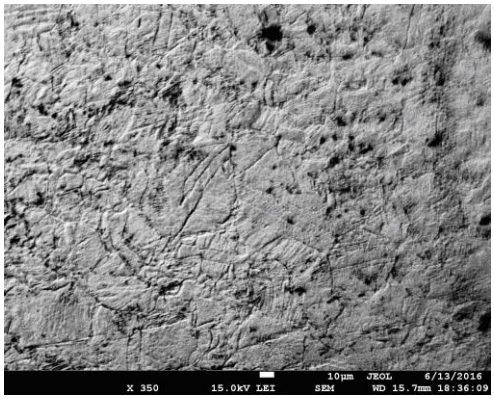

Fig. 5 *Topography of brass CuZn30 surface from SEM, a lower SE detector – magnification 350*

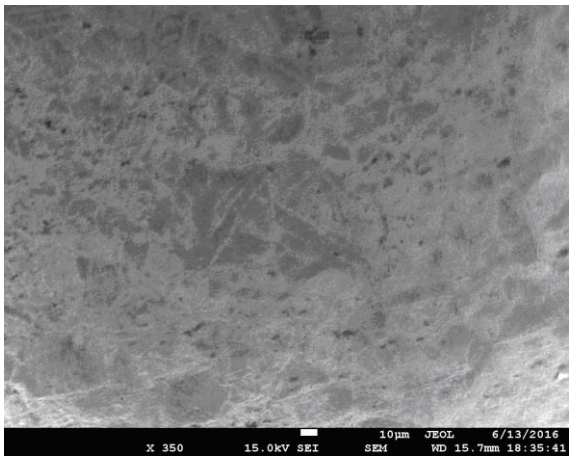

Fig. 6 *Topography of brass CuZn30 surface from SEM, an upper SE detector – magnification 350*

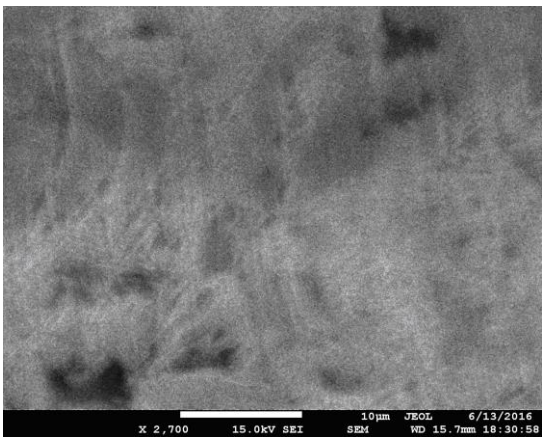

Fig. 7 *Topography of brass CuZn30 surface from SEM, an upper detector - magnification 2700*

3.3 The AFM measurements

AFM measurements were made on Asylum Research MFP-3D Infinity equipment. This equipment contains many various functional modes. Contact mode is good for investigation the topography height profile. Taping modes are suitable for scanning of interactions of the sample surface atoms with the cantilever tip. These interactions modify the tip oscillations. The oscillations are recorded and evaluated from various points of view, such as the change in oscillation amplitude (amplitude retrace), phase shift between the driving signal and mechanical vibrations of the tip (phase retrace) and height profile (height retrace). The beneficial results we can obtain are information about the surface components distribution of multi-component materials from the tapping mode measurements which are the phase and attenuation amplitude images. Time-varying interaction forces between the vibrating tip and the sample contain detailed information about the elastic, adhesive, and dissipative response of the sample. The phase images correlate to the distribution of surface stiffness (Young's modulus) of the material. AFM can be used to reveal subatomic structures. High spatial resolution imaging of material properties is an important task for the development of nanomaterials.

In order to compare the options of AFM with optical and electron microscopy, we made sample surface images in AFM contact mode. Fig. 8 shows the topography of the surface of a sample area of 50 x 50 μm^2. There are visible boundaries of crystal grains and the details in the interior of grains in this picture. The altitude profile of an

area of the surface is well viewable in 3D image in Fig. 9. Fig. 10 and Fig. 11 show the segment of 10 x 10 μm^2 (marked in Fig. 8) where more structure details can be seen.

More interesting information about the surface structure are obtained at measuring in AFM AC taping mode. The segment 2 x 2 μm^2 was chosen in plate portion of the structure, wherein the profile height differences are less than 200 nm. For larger height differences the measurements are burdened by big failures.

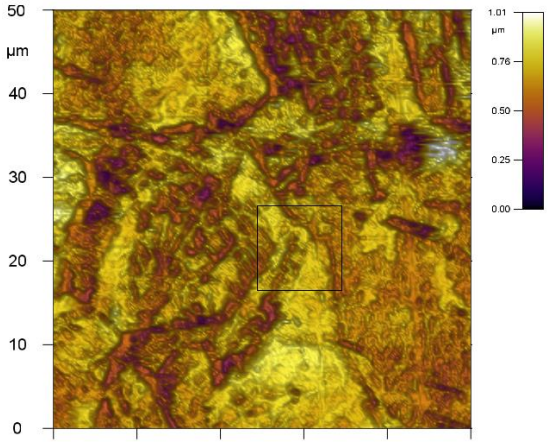

Fig. 8 *Topography of brass CuZn30 surface from AFM in contact mode*

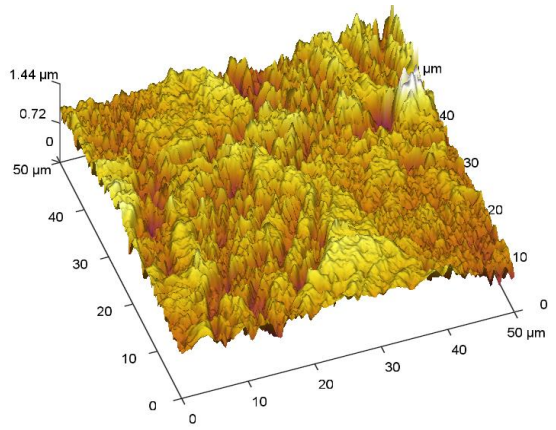

Fig. 9 *3D image from the same area as in Fig. 8 in AFM contact mode*

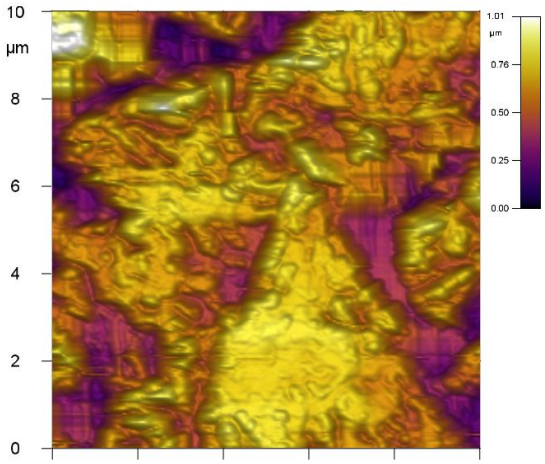

Fig. 10 *Topography of brass CuZn30 surface from AFM \ in contact mode, detail 10 x 10 μm^2 marked in Fig.8*

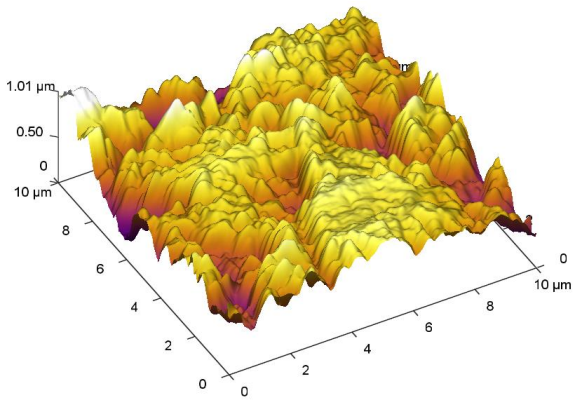

Fig. 11 *3D image from the same area as in Fig. 10 in AFM contact mode*

The Fig. 12 shows the surface image of the segment 2 x2 μm^2 obtained by measurement in AC mode. It is the elevation profile – height retrace. The surface roughness is near the 200 nm. The view of the same area in the decay amplitude (amplitude retrace) is shown in Fig. 13. This measurement is already affected due to increased surface roughness, which results in an asymmetric response when passing through the vertical interface.

Strong signal response is when displaying phase characteristics shown and Fig. 14. The major changes in the phase response of the interaction of the tip and the atoms of the structure are well apparent in the 3D image on Fig. 15. The dark spots on Fig. 14 illustrate the areas with the structural modifications caused by crystallographic defects, segregated impurities and other structural changes. The images of phase signal reveal uneven density of precipitates in grains. The larger phase shift is observed, the stronger interaction it means. This indicates that the black areas will have a different composition and properties, in contrast to bright spots probably solid solution Zn in Cu, black spots – probably precipitates, what is unexpected for this relative pure material. They can be segregated elements present in the alloy as an impurity. The density and size of that is locally varied. An important aspect of this view is that we are able to see the details of the structure in the submicron scale.

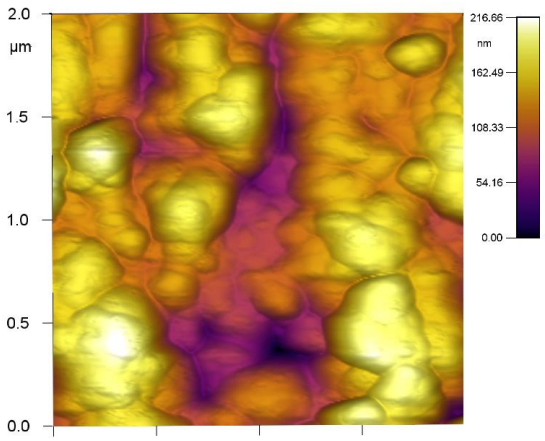

Fig. 12 *A detail 2 x 2 µm² of brass CuZn30 surface in AC taping mode – height retrace*

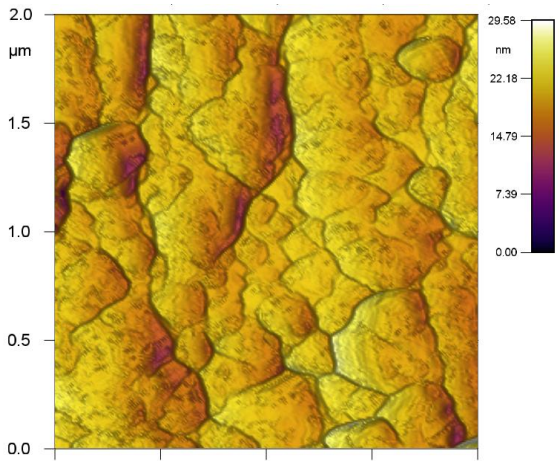

Fig. 13 *The same area as in Fig. 12 in AC taping mode – amplitude retrace*

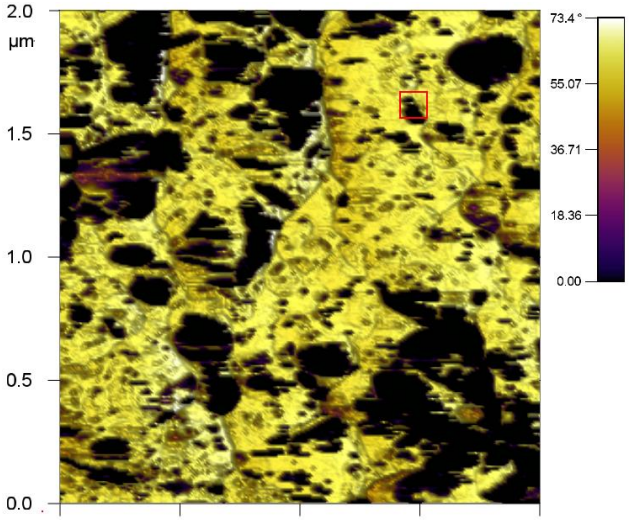

Fig. 14 *The same area as in Fig. 12 in AC taping mode – phase retrace*

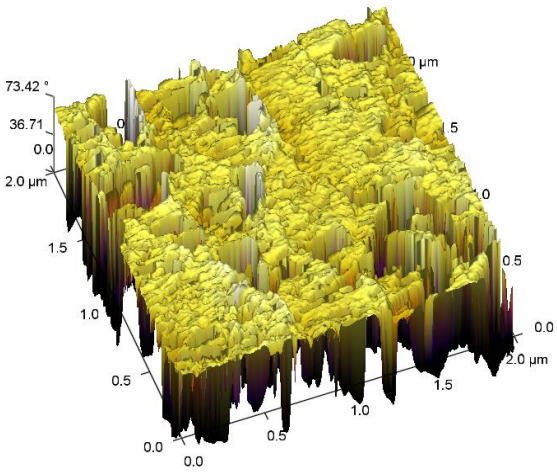

Fig. 15 *3D image of the same area as in Fig. 14 in AC taping mode – phase retrace*

The AFM is a diagnostic method allowing by comparatively simple tools to work in the sub nanometer region. An example of such measurement of the surface segment size 128 x 128 nm² is shown in Fig. 16. It is the amplitude retrace characteristics of the area indicated by the small square in Fig. 14. A strong signal in amplitude characteristics is present at interface of large phase changes, as it is seen from 3D phase characteristics shown in Fig.17. Small spots in Fig. 16 represent the individual location with size less than one half of nm. These spots

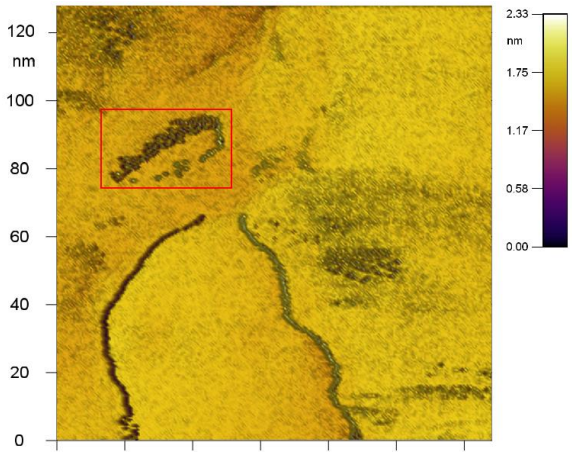

Fig. 16 *A detail 128 x 128 nm² from area marked in Fig. 14 in AC taping mode – amplitude retrace*

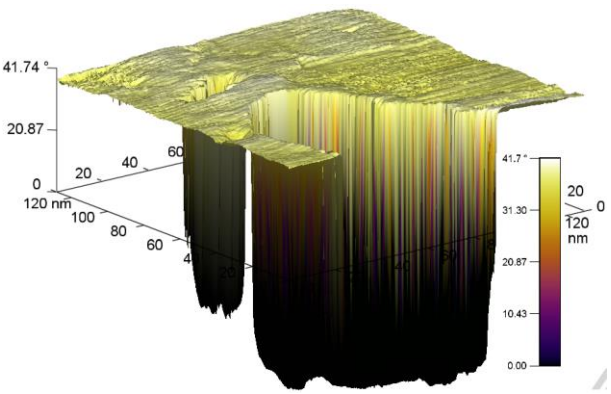

Fig. 17 *3D image of the same area as in Fig. 16 in AC taping mode – phase retrace*

should be individual atoms of the impurities or dislocations. Dark spots on Fig. 16 are the pick signals generated as one pixel pick signal with positive or negative value (in amplitude characteristics) and mutual distances between picks are less than one nm. The signal picks are seen in Fig. 18 and map of signal picks is shown on Fig.19.

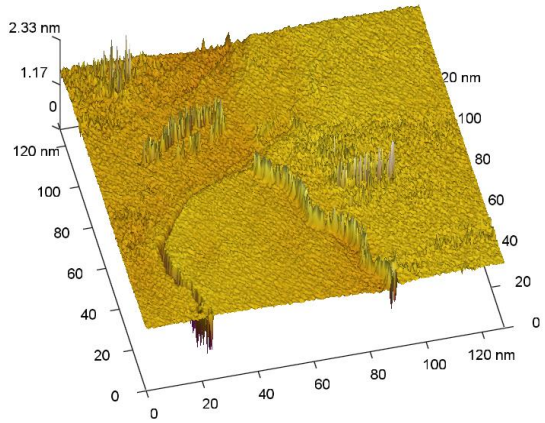

Fig. 18 *3D image of the same area as in Fig. 16 in AC taping mode – amplitude retrace*

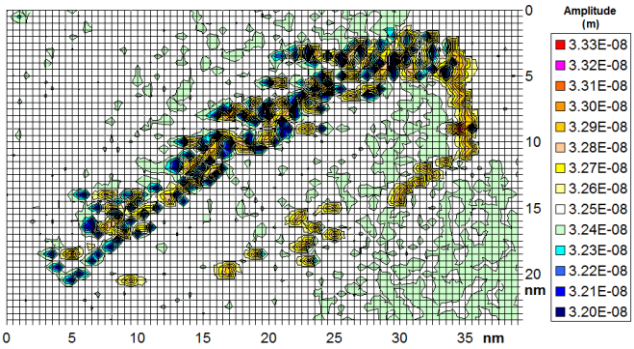

Fig. 19 *Map of individual atoms or dislocations from area marked in Fig. 16*

4 Conclusion

Presented analyses show that using AFM for study of material surface brings much more detailed results than using an optical microscope or SEM. The AFM can distinguish more detailed grain interface because the AFM is able to recognize sub-nanometer peculiarities. Moreover, it is somewhat possible to image the distribution of the individual atoms at the interfaces of different areas what can be useful for next studying of material structure.

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