STUDY OF THE CuZn30 ALLOY MICROSTRUCTURE BY AFM

Erika Hujová, Vladimír Áč, Mária Ličková, Jozef Kasala

Alexander Dubcek University of Trencin, Faculty of Special Technology
E-mail: erika.hujova@tnuni.sk

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1. Introduction

Atomic-force microscopy (AFM) is a type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. AFM 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 [1].

Because a classical optical method is well known in general, we concentrate to introduce AFM, especially one major ability: imaging. 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. The surface topography is commonly displayed as a 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].

2. Configuration of an AFM

An AFM typically consists of the small spring-like cantilever which is carried by the support. Optionally, a piezoelectric element oscillates the cantilever. The sharp tip is fixed to the free end of the cantilever and the detector records the deflection and motion of the cantilever. The sample is mounted on the sample stage.

A cantilever 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. When the tip 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[2]. 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.

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 (AC mode) where the cantilever is vibrated or oscillated at a given frequency [3].

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. 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.

In tapping mode, the cantilever is driven to oscillate up and down at or near its resonance frequency. 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 [4].

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

There were examined a sample of deep-brass: Cu – 70 %, Zn – 30 % in our experiment. The chemical composition of the studied alloy shows Tab.1.

Tab.1. *Indicative chemical composition of the studied alloy CuZn30 (Ms70) 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

From the structural point of view is a single-phase structure composed of a solid solution α. Phase diagram of this alloy is shown on Fig.1. Surface of the sample was cut, polished and chemically etched. Using the etching solution allows to visualize material structure of surface of the sample.

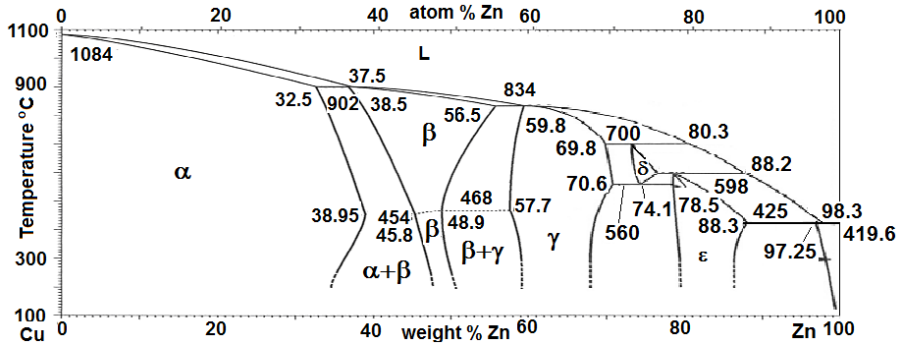


Fig.1: *Phase diagram of alloy CuZn*

Optical microscope and the AFM (Imaging in Contact and AC mode)were used for studying the sample. Both allowed us to survey topography of the sample, especially the grain boundaries (Fig.2). On both pictures, there is clearly noticeable area with the formation twins. Information about altitude profile brings scale heights shown in each picture.

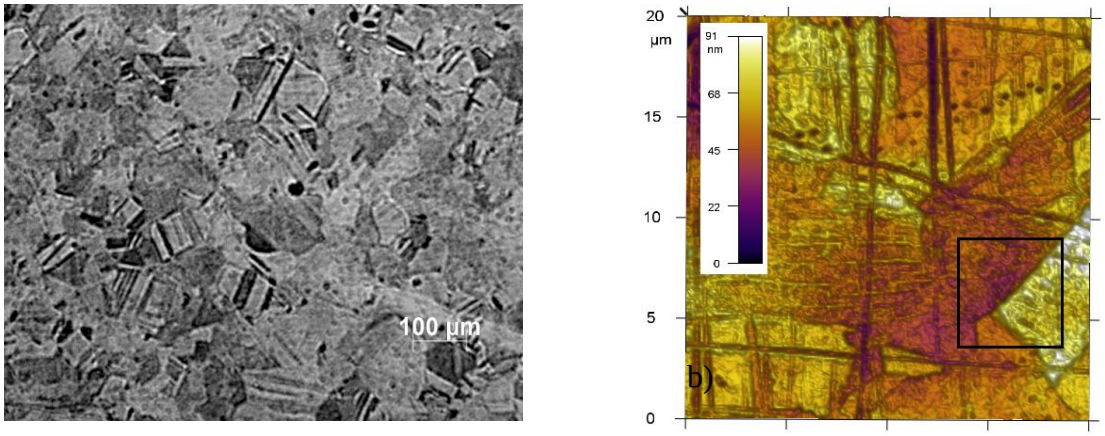


Fig.2: *View of the grain boundaries by the optical microscope (left) and AFM in Contact mode, scan size 20 µm × 20 µm(right).*

For next studying of the sample was used AFM in AC mode with rate of scan 0,2 Hz. We focused our interest in grain boundary between the areas of deformation twinning defined by marked location on Fig.1-right (scan size 5 µm × 5 µm). Obtained image and its detail in marked area are shown on Fig.3.

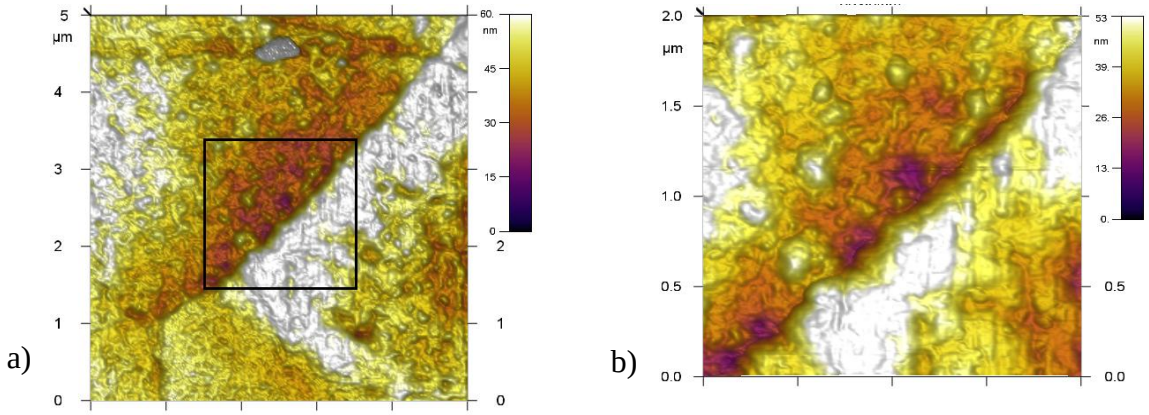


Fig.3: *Height profile of the grains by the AFM in AC mode a) and the detail of grain boundary in marked area b)*

Fig.3 a) shows the height profile of grains, where we can see variation in height of the grain boundaries as a result of selective etching. Using the AC mode offers opportunity to study image of the same scan by the recorded amplitude and phase signals at the same time (Fig.4). The profiles on the Fig.4 copy topography on the Fig.3 b) and the grain shape of the surface.

The amplitude profile (Fig.4, a) brings information about material properties. On Fig.4 a), c), there it is clearly seen that grain boundary has a smaller loss of signal because of structural defects. Grain boundaries are paler due to less amplitude attenuation of the tip - the material is not compact, there are the structural defects. Areas of grains are not homogeneous, the different composition of the grains can be seen.

On the other side, the images of profile of phase signal on Fig.4 b) and d) 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 (bright spots – probably solid solution Zn in Cu, black spots – probably precipitates, what is unexpected for this pure material. They can be segregated elements present in the alloy as an impurity. In one of observed twins on Fig.4b) and d), there is a lower density and larger sizes of precipitates. In the other one, there is the opposite.

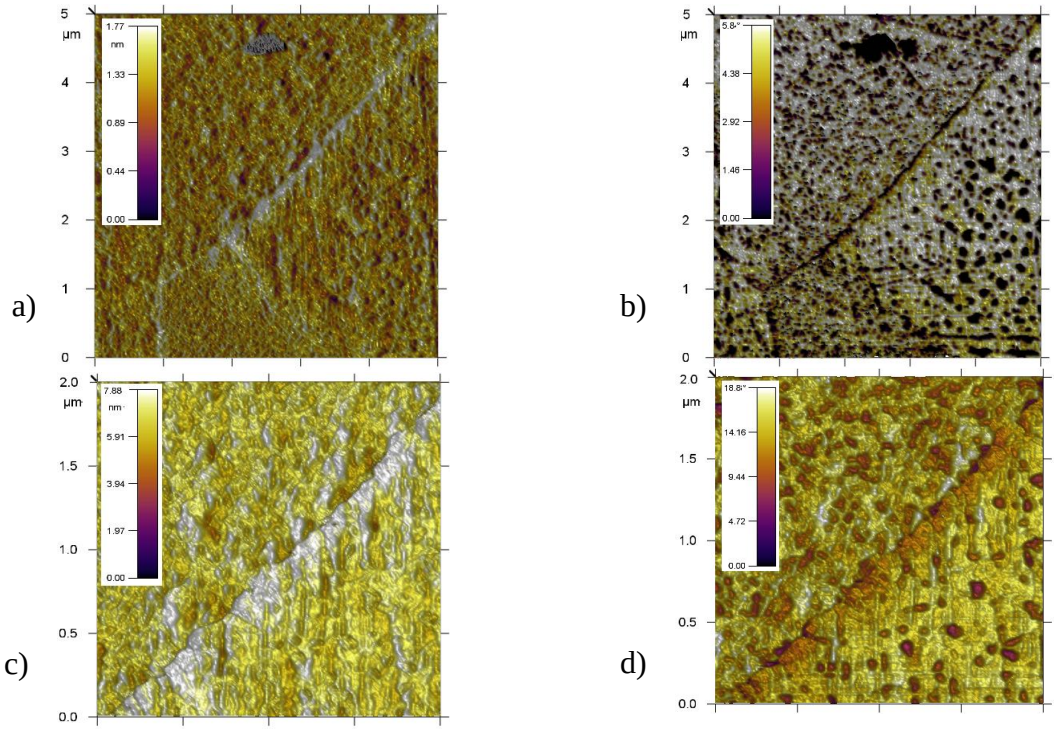


Fig.4: *Amplitude profile a), c) and phase profile b), d) made by the AFM AC mode; scan size 5 μm ×5 μm: a) and b), 2 μm ×2 μm:c) and d)(selected area inFig.3).*

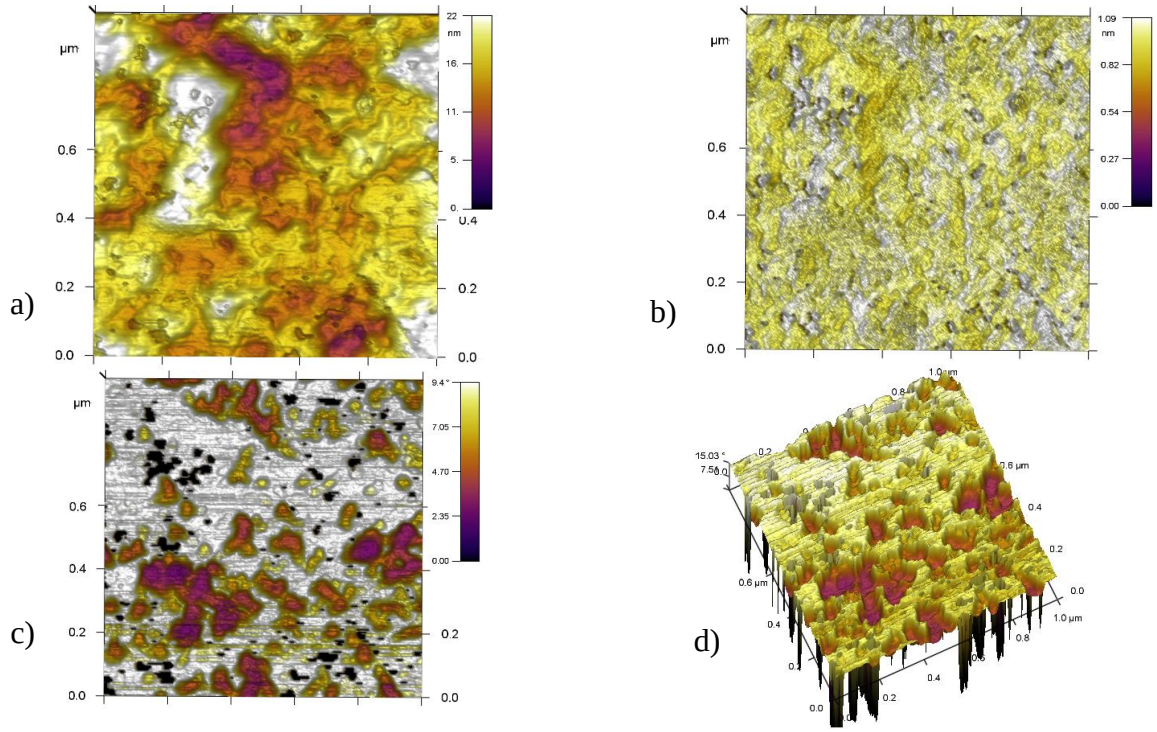
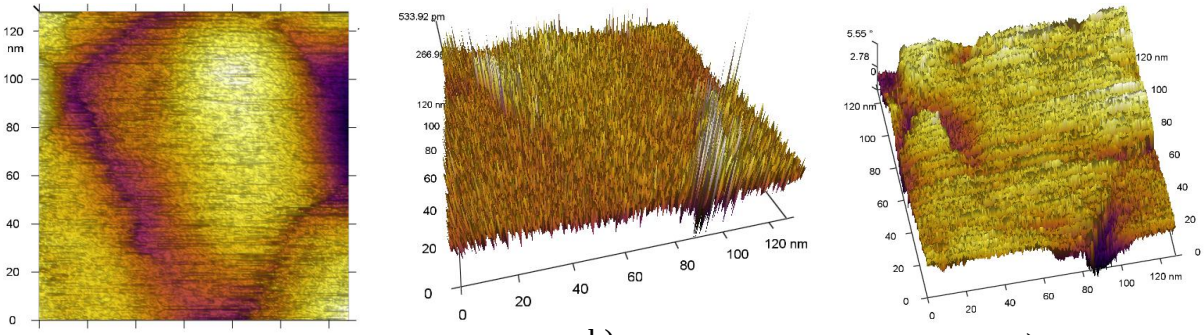


Fig.5:*Detail view in the grain (AFM AC mode; 0,2 Hz),scan size 1 μm ×1 μm: detail the height profile a), detail the amplitude profile b), detail the phase profile c) and its 3D view d)*

Upon further reduce the scanned area (1 μm × 1 μm) can be observed detail the height profile in the grain (Fig.5, a). Similarly, Fig.5 b) shows detail the amplitude profile and detail the phase profile in the grain (Fig.5 c) and d). On the Fig.5 c), there are clearly seen three kinds of bunches of particles with different colour shade: the smallest, larger and the large

stones. Smaller bunches of particles are observable only at this resolution and the best view is in the phase profile. This 3D view in Fig.5 d) highlights the great "phase holes" that belong to the smallest black bunches. Their phase signal depth distinguished these bunches from the others.

Details at the atomic level are presented in the next pictures (Fig.6 a) of larger bunch seen in Fig.5 (amplitude profile). Fig.6 b) shows its 3D view, there are peaks which constitute individual atoms on the grain boundary. Atoms on a compact area cannot be distinguished. They are visible only in the case of clusters of atoms in a homogeneous medium. Phase profile on Fig.6 c) shows the sharp phase response in place of individual atoms.



a) b) c)

Fig.6:*The height profile in the some detail a) and its3D view of amplitude characteristics b) (AFM AC mode; scan size 128 nm × 128 nm; 0,2 Hz), and 3D view of phase profile c)*

4. Conclusion

Presented analyses show that using AFM for study of material surface brings much more detailed results than using an optical microscope. The AFM can distinguish more detailed grain interface because the AFM is able to recognize sub-nanometer peculiarities. Unlike the optical microscope, especially in CuZn30 alloy the AFM allows to see differences in the density and size of bunches in twinning areas, shape and structure of various bunches of particles in basic α-phase – all of these thanks to the amplitude and phase characteristics that reflect the force interactions of surface material atoms. 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.

Acknowledgement

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5. References

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