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Qualitative Analysis of the Large Plastic Deformed Al-Mg Alloy Probe Using Atomic Force Microscopy

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ABSTRACT

AFM is a method used for qualitative investigations of the two probes of Al-Mg alloy. The probes represent two states of the material: 1) initial state, 2) deformed under progressive shearing and cumulating a large strain rate. In the last case, the material is nanostructured, that means the grain size is diminish. The paper deals with the measurement of the grain size, roughness and anisotropy of the material by means of the Fourier analysis.

Keywords: *AFM, nanostructured Al-Mg, mechanical properties*

1. Introduction

Recent development of the new structured materials determines the necessity of using new techniques of investigation at micro and macroscale. The ultrafine grained materials are obtained by acting the materials to the large plastic deformation and the dimension of the grains could rich the dimension of 100 – 50 nm. At this size, the classic microscopy cannot provide enough accurate information, so that the atomic force microscopy could explore the surface in a better way. The mechanical characteristics of the investigated surface are determined indirectly, based on the reaction of the material atoms in report to the atomic force microscopy tips.

The paper proposes the investigation of the two probes of Al-Mg alloy A5182. The difference between the two probes is that the initial one is an undeformed structure, and the second one is obtained by multidirectional shearing which are cumulated and rich to 20% strain rate. That corresponds to a structure that begins to have ultrafine grains. The mechanical characteristics of these two structures are different due to the modification inside the microstructure. It is known that the nanostructured materials exhibit a high hardness that causes a high capacity to avoid corrosion and oxidation..

2. Preparation of the probes

Two probes of the dimension 15 mm x 15 mm where analysed in the central area of 2 mm x 2mm. This inner size is determined by the area where the deformation of the material is cumulated to 20% strain rate due to the shearing applied on the rotated probe with 90 degrees. The probes are deformed on the testing machine MTS 20 using a proper device for shearing in two directions. The probe was rotated progressively with 900, to reach 20% of strain rate. This strain rate correspond to a plastic state of the material.

Following rotation, the area of the probe that exhibit a cumulated strain rate of 20% is only of 2 mm, as presented in figure 1.

3. Principle of AFM measurement

In this paper we analyzed surfaces with a high density using the Nanosurf AFM with the measuring principle described in following.

An Atomic Force Microscope (AFM) is made up of a sharp tip mounted or inserted intoone end of a very small cantilever, which is

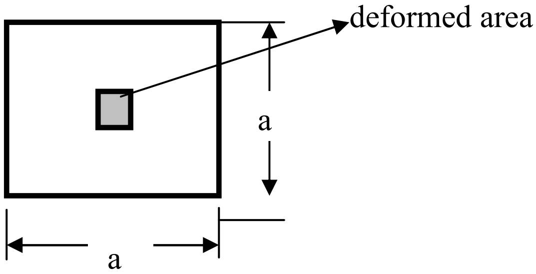


Figure 1 – The shape and the size of the probe undergo to shearing with 2% strain rate per cycle. 10 cycles of the shearing were carried out by rotating the probe and clamping in the testing machine device. a=15 mm, thickness of 2 mm

mechanically moved over the surface to be examined. The variations of the surface height affect the force acting on the tip and therefore vary the bending of the cantilever. This bending is measured and recorded line by line.

Dynamic mode: In dynamic mode, the tip is not in contact with the surface. The cantilever is oscillated at its resonance frequency, which is influenced by the forces coming from the sample. The frequency deviation is used to make an image of the sample.

An important parameter to be quantitatively analyzed in all microscopy techniques is the spatial resolution. AFM produce three-dimensional images of the surface topography and the resolution strongly depends on the tip geometry. In order to reduce the geometric convolution of the surface, tips with high aspect ratios and low effective radii are required

4. Results and interpretation

4.1. 3D representation of the surface investigated

The data obtained from the Nanoscan software that works together with the AFM, set up for dynamic mode, are imported in the statistic analysis software SPIP that generates the graphs of the surface topography (figure 2). The roughness is also measured in a same graph where the waviness of the surface is superposed. The profiles obtained for the two probes are presented in figure 2 and figure 3.

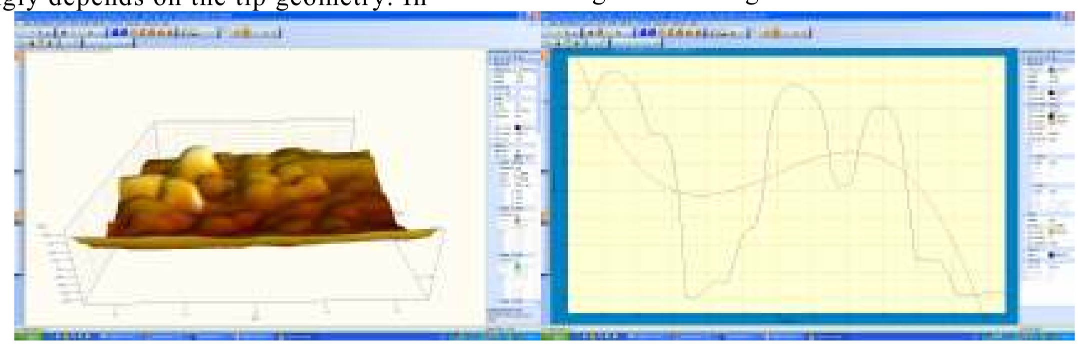


Figure 2 Topography of the surface of the Al-Mg under 20% strain rate
5.0 μm x 5.0 μm x 238.8 nm
a) 3D representation of the investigated surface, b) roughness of the surface

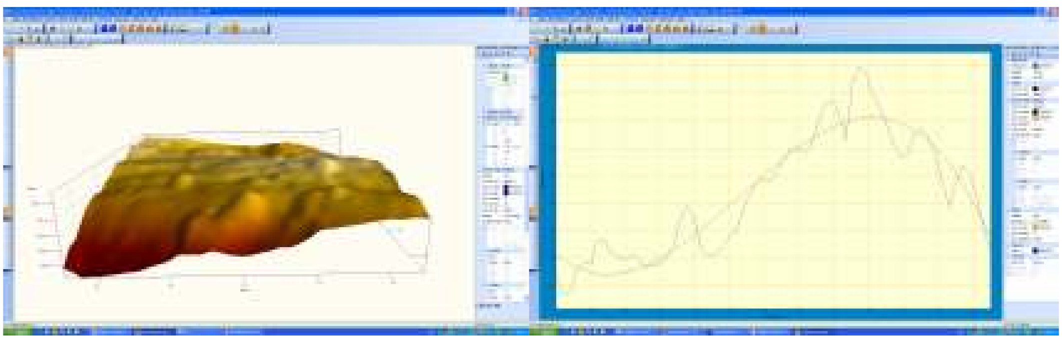


Figure 3 Topography of the surface of the nanostructured Al-Mg under 20% strain rate
5.0 μm x 5.0 μm x 222.2 nm
a) 3D representation of the investigated surface, b) roughness of the surface.

As it could be noticed in the figures below, the roughness in the case of the nondeformed probe is greater then in the case of the nanostructured material. The arrangement of the atoms and molecules during large plastic deformation determine an alignment of the structure that gives new properties to the material. In the figure 3a by comparison with figure 2a the height of the hills visible in the 3D representation are represented by the grains. In figure 3 the size of the hills – the grains – are smaller then in the figure 2. As a consequence, the roughness of the surface is diminishing in the case of the nanostructured material.

4.1. Measurement of the grain size

AFM allows to identify and isolate the grains on the investigated surface and to measure their size. In figure 4 and 5 there are the representation of the area where the grains were measured and the white line indicates the area where the grains were considered.

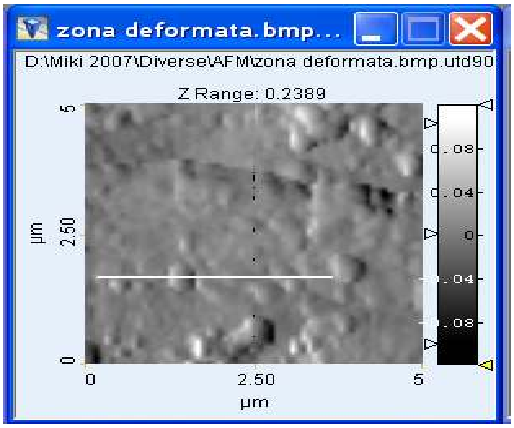


Figure 4 - Measurement of the grain size using ISO5436 Step Height method – deformed probe

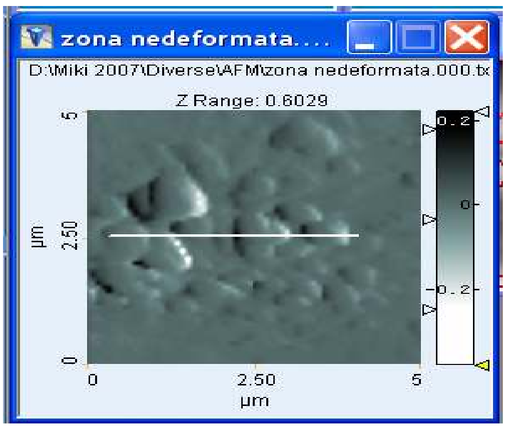


Figure 4 - Measurement of the grain size using ISO5436 Step Height method.

The smallest grain size present in the microstructure of the probe is 420 nm for the nondeformed structure and 20 nm for the for nanostructured material for the reference height of 180 µm.

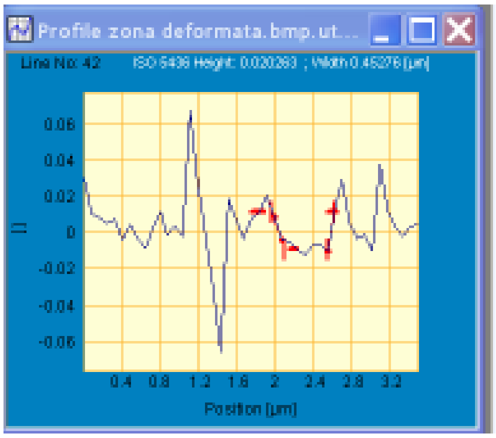
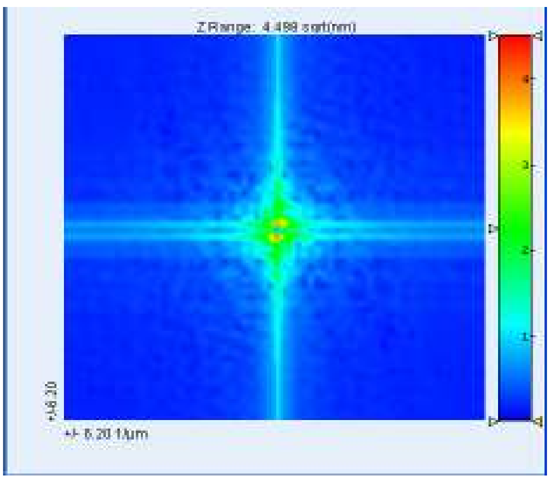


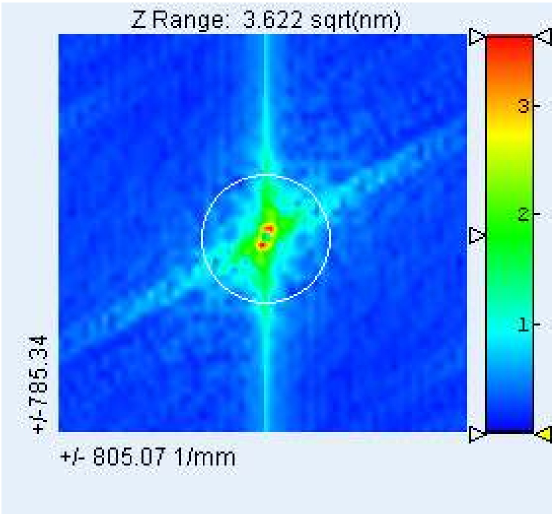
Fig. 5 – Measurement of the grain size using the profilogram Image processing SPIP software

4.3. Measurement of the anisotropy

The effect of the nanostructuring of the material under large deformation is noticed by the dimension of the grain in the case of the deformed probe. The size of the grain is 20 nm and the initial grain size 420 nm. This is a qualitative measurement because it is an indirect method to obtain the grain size. To prove this conclusion, a SEM (Scanning Electronic Microscopy) analysis will be done on the ongoing researches to measure the grain size inside the material.



a)



b)

Fig. 6 The two-dimensional Fourier Transformation (FT) for the nondeformed probe (a) and for the deformed probe (b)

In Fig. 6, the two-dimensional FT's are shown at the right of each image. Peaks (bright) in the 2D-FT represent the intensity of frequency components in the surface image. The frequency domain ranges from $-(1\Delta)-1$ to $+(4\Delta)-1$ where Δ is the sampling interval (in our case, the AFM scan step). For a 2D image we have Δx and Δy. In Fig. 1d the Fourier disk is small and slightly elliptic since the surface structures (mainly dots) are not well resolved, making the shapes more anisotropic than they actually are. For figures below, the 2D-FT is more symmetric and the disk radius is a bit larger, indicating that higher spatial frequencies were measured and improved lateral resolution of the nanostructure shapes was obtained.

The presence of a greater anisotropy in the case of the deformed probes, contrary to the expectation that an arranged atoms structure will give us a small anisotropy, could be explained by the presence of the shear bends in the case of aluminium alloy. It is known that aluminium exhibit shearing walls that are shown also in the 3D image of the investigated surface.

Local anisotropy is smaller in the case of the deformed probe, but globally it is an increasing of the anisotropy. This effect is probably eliminated in the case of other materials like Ti, Mg.

5. Conclusions

The quantitative evaluation of the contrast of dynamic AFM images in order to determine local material properties is still a challenge. The contact resonance spectroscopy allows one to deduce mechanical parameters such as the indentation for hardness and shearing modulus from flexural and torsional contact resonance frequencies provided that low enough excitation amplitudes are used permitting to linearise the interaction force.

6. Acknowledgements

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Analiza calitativă cu ajutorul microscopiei cu forță atomică a unor probe de aliaj de Al-Mg supusa deformațiilor plastice mari

Rezumat:

Microscopia cu forta atomica este folosita in investigarea calitativa a unor probe de aliaj de Al-Mg. Probele reprezinta materialul in stare intiala si in stare deformata obtinuta prin aplicarea unor incarcari corespunzatoare forfecarilor succesive. In aces caz, materialul este nanostructurat, ceea ce inseamna ca marimea grauntilor este determinata. Lucrarea trateaza rezultatele obtinute de la investigarea AFM a acestor probe, in cazul imaginii 3D a suprafetelor, rugozitatii, marimii grauntilor si a anisotropiei obtinute prin intremediul analizei Fourier

L'analyse qualitative des grandes déformations d'alliage Al-Mg en utilisant la microscopie de force atomique

Résume:

AFM est une méthode utilisée pour l’ investigation qualitatives des deux éprouvettes d'alliage d'Al-Mg. Les éprouvettes représentent deux états microstructurales different dans la materielle: 1) l'état initial, 2) déformé en cisaillement progressive telle que’une grand taux d'effort est accumulée. Dans le dernier cas, la materielle est nanostructurée, qui signifie que la taille des grains sont diminuent. L'article présent la méthode de détermination de cette taille du grain nanostructurée, l'âpreté et l'anisotropie de la microstructure en utilisant l'analyse Fourrier