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TOPOGRAPHY STUDIES OF A MAGNETRON REACTIVE SPUTTERED COATING

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The present paper shows changes of the morphological aspects of the samples surfaces due to different methane concentrations in a magnetron deposition chamber. Using 5 cathodes, Cu, Si, Ti, Y and Zr and a C45 substrate, the methane flux in the chamber was set at 35% and 50%. The samples surfaces are studied using an atomic force microscope.

Keywords: magnetron sputtering, surface morphology, atomic force microscopy, vertical build-up, lateral growth

1. Introduction

High entropy alloys, HEAs, are currently intensely studied. Until now bulk metallic glasses, BMGs, were the main research direction. Developed slightly later, HEAs were initially less studied than BMGs, and recently a new concept of HE-BMG (high entropy bulk metallic glass) allows a comparative study on the alloy design perspective [1].

HEAs consist of 5 to 13 elements in equiatomic or near equiatomic ratios, with excellent properties: mechanical, optical etc. [2].

To obtain such alloys magnetron sputtering is usually the preferred technique, using inert gas ions, Ar in our case. Sputtering is defined as surface removal of atoms by energetic ions [4, 5, 6].

After several theoretical estimations the magnetron setup used 5 cathodes, Cu, Si, Ti, Z and Zr, and from a gas atmosphere, CH_4, we introduced C by reactive sputtering, in order to obtain a 6 element coating.

2. Experimental part

2.1 Sample preparation

The coatings are obtained by magnetron sputtering using the above metioned cathodes having 50 mm in diameter, each with a minimum purity of

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99.95%. The distance bewteen cathodes was 170 mm, the deposition temperature 300ºC and a C45 steel as substrate.

Prior to the deposition the substrate surface was cleaned for 5 min by sputtering with Ar ions. These parameters are kept constant for each sample, the CH_4 flux was solely modified, at 35% and 50%.

For ease of identification we have adopted the following notations:

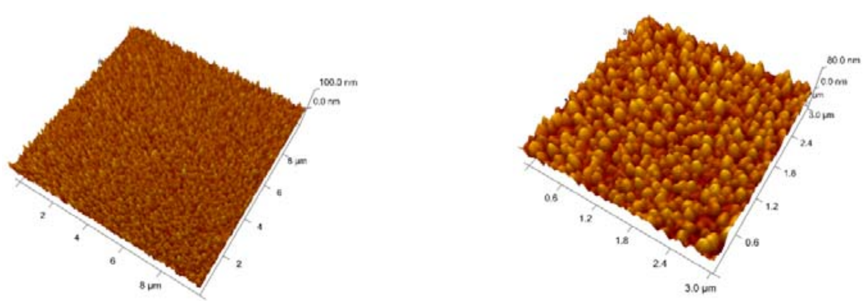
- (CuSiTiYZr)C35 for the sample obtained at 35% CH_4 flux
- (CuSiTiYZr)C50 for the sample obtained at 50% CH_4 flux !!!! verificati mai bine articolul inainte sa-l trimiteti la recenzie !

2.2 AFM investigations

The obtained samples are investigated using an atomic force microscope Innova – Veeco in tapping mode. The investigated samples are of 100µm^2 and 9µm^2. On both surfaces the roughness, the size and distribution of the particles (sau alt cuvant, formations nu merge) were determined, as well as several profile lines were examined.

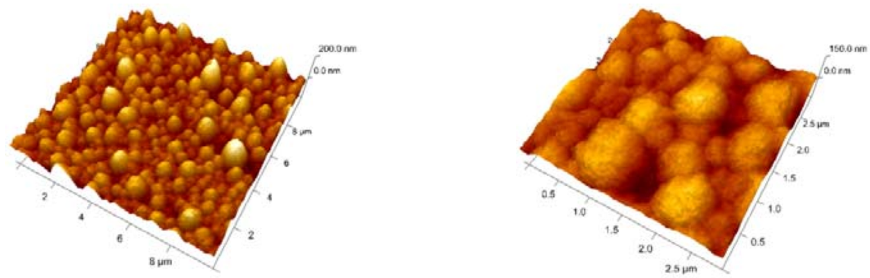
3. Results and discussion

Figure 1 and 2 depict the investigated surfaces of 100µm^2 and 9µm^2. For both materials dome shaped structures are to be observed, uniformly distributed on the entire surface. When increasing the CH_4 flux taller domes with an irregular distribution are obtained, shown in Figure 2.



a. 100µm^2 b. 9µm^2

Fig. 1 AFM topography of (CuSiTiYZr)C35



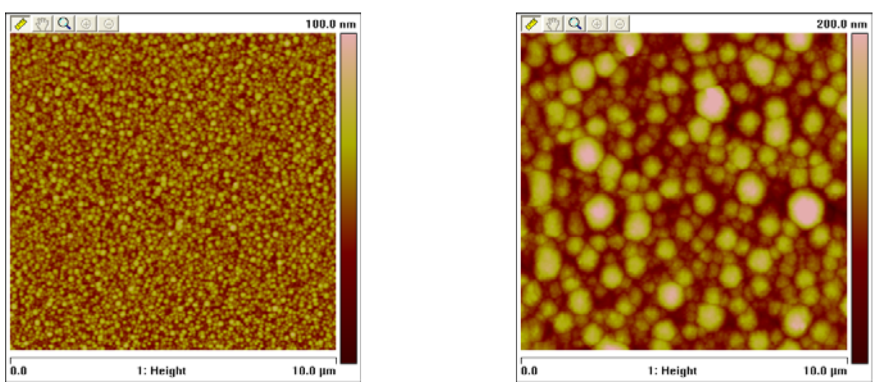
a. 100µm^2 b. 9µm^2

Fig. 2 AFM topography of (CuSiTiYZr)C50

The competition on vertical growth and lateral diffusion clearly influences the morphology of the grown surfaces. Vertical growth occurs due to the accumulation of atoms impacting from different incident angles because of sample rotation. The growth kinetic is governed by the mobility of the incident atoms on the surface prior to entrapment in the layer. This mobility may be enhanced by an energy surplus.

The growth of thin films is usually explained by three models: Volmer – Weber, Frank – Van der Merve or Stranski – Krastanov [7, 8]. The last model suggests the formation of one or several mono-layers, thus further growth is hindered. Spheroidal particles are formed. This growth mechanism is observed for metal - metal or metal - semiconductor systems.

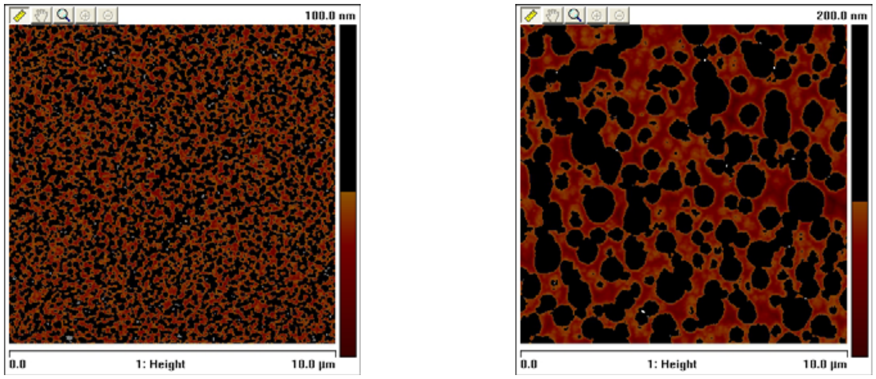
In order to analyze the particles surface distribution two top views of the 100µm^2 topographies are shown in Figure 3.



a. (CuSiTiYZr)C35 b. (CuSiTiYZr)C50

Fig. 3 Top view of the original surfaces

To discriminate dome surfaces an approximate medium height was selected, the specified region of interest being subtracted for further investigation, as Figure 4 shows.

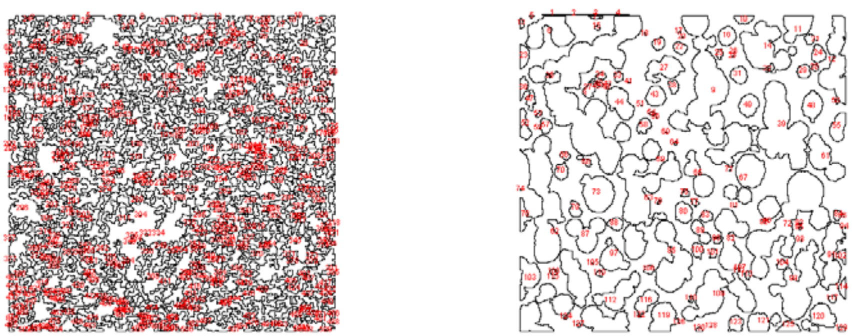


a. (CuSiTiYZr)C35 b. (CuSiTiYZr)C50

Fig. 4 Subtracting the regions of interest

The surfaces above the specified height were analyzed. A count was performed on Figure 5, followed by the determination of the mean region of interest area.

For (CuSiTiYZr)C35 a total number of 509 surfaces are counted, while for (CuSiTiYZr)C50 126, while mean areas of 0.09µm^2 and 0.42 µm^2 are calculated. The standard deviation of the surface areas was 0.41 for the first and 1.00 for the second indicating that the 35% flux sample particles are more uniform in size, when compared to the 50% flux which indicated taller domes unevenly scattered across the surface.



a. (CuSiTiYZr)C35 b. (CuSiTiYZr)C50

Fig. 5 Investigated surfaces contours

For the (CuSiTiYZr)C35 sample the region of interest covers 49.69 µm^2 of the entire surface, while 52.42 µm^2 for the (CuSiTiYZr)C50.

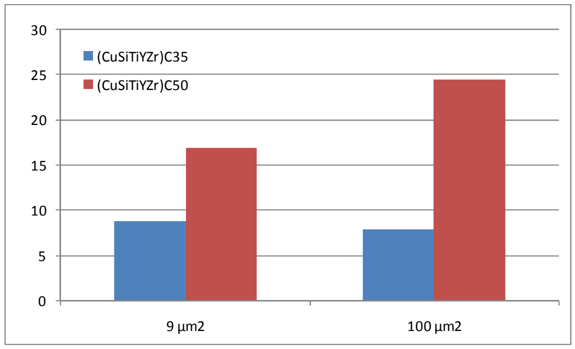


Fig. 6 Surface roughness variation

The surface roughness on both investigate surfaces exhibited in Figure 6 indicates a lower value for the sample prepared at a lower methane flux, (CuSiTiYZr)C35.

4. Conclusions

In order to evaluate the influence of the methane flux in the deposition chamber on different samples surface morphology, a 35% and 50% flux were chosen.

Dome shaped particles are observed on both surfaces, but at a larger methane flux these domes are larger in diameter and more pronounced, resulting in a broader surface.

The number of identified formations on an investigated area was 4 times greater at a lower methane flux.

The region of interest area calculated for both cases showed close values, but the surface of individual formations indicated a large variation from particle to particle.

The distribution on the surface was random with no preferential growth region, which is a consequence of substrate rotation during deposition.

The higher methane flux indicated a greater value for surface roughness. The surface morphology associated with several line profiles investigations indicated a best fit to Starski - Krastanov growth mechanism.

Since the morphology of the growing surfaces is strongly determined by the competition between vertical build-up and lateral diffusion, a further analysis of the deposition parameters in order to improve the surface topography is needed.

The vertical build-up is a result of the columnar particles obtained because of different atoms incident angles, while the lateral growth depends on surface diffusion, mainly determined by the kinetic energy of the incident atoms.

In order to improve the surface topography at a higher methane flux the magnetron configuration needs to be modified in order to assure a higher kinetic energy for the incident atoms.

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R E F E R E N C E S

[1] *Sheng GUO*, et al/Progress in Natural Science: Materials International 21(2011) 433−446
[2] *YEH J W*. Recent progress in high-entropy alloys [J]. Annales De Chimie-Science Des Materiaux, 2006, 31: 633−648
[3] *Yeh et al*, Novel Alloy Design Concepts and Outcomes, Advanced Engineering Materials, 2004, 6, No. 5, DOI 10.1002/adem.200300567
[4] *Peter Sigmund*. Theory of sputtering. I. Sputtering yield of amorphous and polycrystalline targets. Physical Review, 184(2):383–415, 1969
[5] *J. Bohdansky*. A universal relation for sputtering yield of monoatomic solids at normal ion incidence. Nuclear Instruments and Methods in Physics Research, B2:587, 1984
[6] *Y. Yamamura and H. Tawara*. Energy dependence of ion-induced sputtering yields from monatomic solids at normal incidence. Atomic Data and Nuclear Tables, 62(2):149–253, 1996
[7] *M. Ohring*, The Materials Science of Thin Films, Academic Press, 1992, ISBN 0-12-524990-X
[8] *K. N. Tu, J. W. Mayer, and L. C. Feldman*, Electronic Thin Film Science for Electrical Engineers and Materials Scientists, Macmillan, 1992. Originally published in IBM J. Res. Develop