Characterization of large area nanostructured surfaces using AFM measurements

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Abstract

A surface characterisation study has been developed to validate an innovative tool making solution for nano patterning large areas via anodizing of aluminium (Al) and subsequent nickel electroforming. A surface topography characterization through atomic force microscopy (AFM) indicated a decreased magnitude of the 3D surface amplitude parameters chosen for the analysis, when increasing the Al purity from 99,5% to 99,999%. AFM was then employed to evaluate the periodical arrangements of the nano structured cells. Image processing was used to estimate the average areas value, the height variation relative to an average plane and the coefficient of variation of the fitted features curvature radius.

1 Introduction

The establishment of nano patterned surfaces influences physical property of a surface such as colour appearance, hydrophilicity or hydrophobicity resulting in self-cleaning effects and great improvement of surface-based bio-analysis. However, the establishment of micro and nano patterning surfaces technologies remains exploratory and commercialization is delayed by the lacking of mass fabrication processes. Moreover, in order to validate and control the quality of the manufactured components with critical dimension in the nanometre range, metrology becomes challenging. For this reasons, the present paper describes a measuring strategy based on AFM measurements and introduces a relocation strategy enabling reproducibility over two different 30x80 mm^2 nickel nanostructured surfaces. The first analysis focused on scanning areas of 15x15 μm^2 and was carried out comparing conventional amplitude parameters as Sa, Sdr and the Abbott curve on both nickel substrates: electroformed Ni made from 99,5% and 99,999% Al substrates. Secondly, a nano

geometrical characterisation of the highly packed and regular structured nickel surface generated from the 99,999% Al substrate was conducted.

Experimental set up

Nanometre-sized structures were fabricated via anodizing two Al substrates with two different purity levels, 99,5% and 99,999% respectively. The Al sheets were cleaned, degreased and then annealed (550ºC for 3 hours) before being electropolished. In the anodizing treatment the surface of the Al plates was converted into a thin oxide film by dipping the plates in a bath containing an acid electrolyte. The porous oxide layers were obtained on both Al substrates and then dissolved in etchant solutions. Afterwards, the obtained pseudo-hexagonal templates were replicated by nickel electroplating with controlled process parameters, to avoid defects such as high surface roughness, pitting, poor adhesion and high internal residual stresses. Finally, the Al substrates were dissolved to reveal the final topography on the nickel surface (see Figure 1).

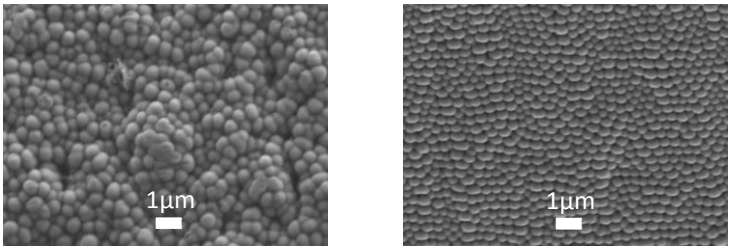

Figure 1: SEM pictures of the two nickel substrates. Left: nickel structure resulted from Al 99,5%. Right: nickel structure resulted from Al 99,999%.

3D analyses of surface topographies were carried out by AFM measurements performed in contact mode, and subsequent image processing through the scanning image processor software [1]. Precise relocation was ensured in order to compare measuring results obtained on different samples. The measuring strategy involved a sample stage (responsible of keeping the sample in place during each measurement) and a coordinate measuring machine (CMM) on which the AFM was mounted to allow surface characterization on large areas (X × Y × Z max 400 mm × 100 mm × 75 mm) [2]. The linear encoder mounted on the X-axis of the CMM enabled measuring steps with resolution of 1 nm whereas movement on the Y-axis was locked in a predetermined position.

2 Surfaces characterization

Measurement reproducibility based on the proposed measuring strategy was tested. The nickel master surface was measured and removed from the stage. 24 hours later it was mounted and measured again. Measurements of the reconstructed images were performed and repeatability of the relocation procedure was evaluated. As a result, the standard deviation between Sa average values of three different measurement repetitions on the same spot for each day was equal to 0,4 nm. A quality control comparison between the two structured nickel surfaces is reported in Table 1. In particular, mean values of roughness average Sa and functional parameters (Sk, Spk, Svk) calculated by accumulation of the height distribution histogram and subsequent inversion [1], show lower values for the Ni-Al 99,999%. Same level of average values were calculated for the hybrid parameter Sdr which calculates a percentage of additional area contributed by the textured surface compared to an ideal plane.

Table 1: 3D data analysis, of both nickel surfaces electroformed from the Al 99,5% (Ni-Al 99,5%) and the Al 99,999% (Ni-Al 99,999%) respectively, (average values are indicated with their experimental standard deviation over 3 repetitions).

3D surface amplitude parameters	**Ni-Al 99,5%**	**Ni-Al 99,999%**
Sa [nm]	98±1	38±2
Sdr %	11±1	12±1
Sk – Spk - Svk [nm]	304±2 - 104±2 - 127±1	131±5 - 34,4±2 - 39,3±1

The more regular and levelled self-organized pseudo-hexagonal array, produced employing the high purity Al substrate was further characterized. In order to quantify the influence of the different processes on the final surface topography responsible for its final functionality new parameters were proposed. The analysis was based on the evaluation, over a scanning area of 15x15 μm^2, of the height variation of 50 randomly selected structures with respect to a reference plane obtained by correcting the global image with a first order least mean square fit algorithm. Mean values of the average height was equal to 72 ± 40 nm. Moreover apex curvatures of 50 more randomly picked nickel bumps were fitted and the corresponding radius estimated. The coefficient of variation (standard deviation/average feature value) for an average

radius value of 330 nm, describing the radius of curvature distribution, equal to 19% was calculated. Finally, watershed segmentation [1] allowed the detection of the texture cells and their characterization trough the so-called features parameters [3]. In particular an average area value of 0,27±0,1 μm^2 was calculated above the same evaluation area of 15x15 μm^2.

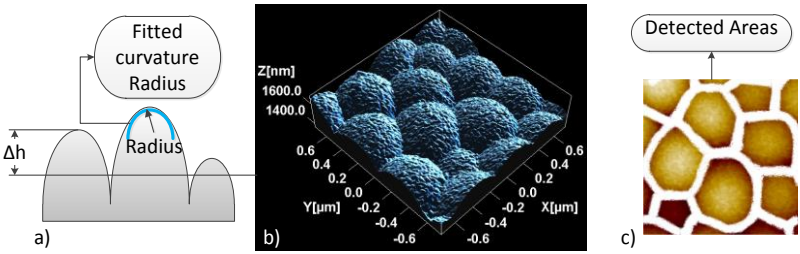

Figure 2: a) Measurement technique to estimate height variation and radius of the fitted features curvature; b) example of 1,5x1,5 μm^2 nickel scanned surface: c) texture cells detected on the nickel substrates used to calculate the average area.

3 Conclusion

The different Al composition resulted on more refined nickel structured surface when increasing the Al content from 99,5% to 99,999%, creating a more homogeneous periodical arrangements of the nano structure cells. The calculated standard deviations could be used as reference parameters to characterize and quantify the stresses behaviour of the oxide layer formation during anodization. The proposed measurement strategy represents a potential tool to improve the geometrical characterisation and quality control of large nanostructured surfaces into a production environment.

Acknowledgment :

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References:

[1] Image Processing Software for Microscopy (SPIP), http://www.imagemet.com/.
[2] H.N. Hansen, N. Kofod, L. De Chiffre and T. Wanheim, Calibration and Industrial Application of Instrument for Surface Mapping based on AFM, CIRP Annals - Manufacturing Technology, 2002, Volume 51, Issue 1, pp. 471-474.
[3] ISO-25178-2, ISO/DIS 25178-2 - Surface texture, 2010.