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Antireflection coating of microstructures by anodizing and influence on the dimensional accuracy of structures

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Abstract
Effective micro- and macro-optical sensory constructions require an anti-reflective coating to prevent internal stray light. Otherwise the scattered light leads to measurement noise, false signals or sensor saturation affecting the functionality.Optical apertures in the range of d = 300 µm and less, which can be prepared as pinhole or slit by UP-machining, have high demands in the single-digit micrometre range on roundness and dimensional accuracy to avoid aberrations. The structural quality should not be influenced adversely during the coating process. One possible, and with less R = 4 % reflectivity in the visible range, competitive coating is black Al_2O_3 anodization. Due to its ceramic fabric and the bonding to the substrate it is particularly resistant. The layer can be produced directly on ultra-precisely manufactured aluminium (ACP 5080) structures that is suitable for anodizing.The coating process is a sequence of etching in 10 % NaOH, the anodizing step in 17 % H_2SO_4 and a colouration process. Dimensional losses occur in particular in the first two steps. During the etching process there is only a removal of the surface. The surface modification during the anodizing results in a removal with simultaneous layer growth. The superposition of the additive and subtractive contributions defines the total loss. In particular tiny cracks on the edge of bodies in the ceramic material may occur due to the resulting structural weakening. Regarding a compensation of those losses, predictions about the erosion behaviour have to be enabled. Depending on the anodic current, the etching and anodization time, the resulting removal rates can be influenced. However, a reduction in the material removal results in an increased reflectivity. An application-related compromise of both parameters should be aimed. The influence of parameters on these conflicting requirements was examined in order to derive design-related technological recommendations.

Keywords: antireflection coating, micro optics, anodization

1. Introduction

Effective micro- and macro-optical sensory constructions require an anti-reflective coating to prevent internal stray light. Otherwise the scattered light leads to measurement noise, false signals or sensor saturation affecting the functionality [1]. Optical apertures in the range of r = 300 µm and less, which can be prepared as pinhole or slit by UP-machining, have high demands on roundness and dimensional accuracy in the single-digit micrometer range to avoid aberrations.

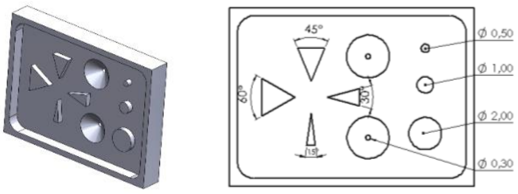

Figure 1. Specimen with different test geometries

The structural quality should not be influenced adversely during the coating process. One possible, and with a reflectivity of less than R = 4 % in the visible range, competitive coating is black anodized Al_2O_3. Due to its ceramic fabric and the bonding to the substrate it is particularly resistant [2]. This method has been established in particular in aerospace applications [3] and extensively investigated in its formation and durability [4, 5]. Alternatively, black nickel electrodeposition can be used [6]. Depending on the anodic current, the etching and anodization time, the resulting removal rates can be influenced. However, a reduction in the material removal results in an increased reflectivity. An application-related compromise of both parameters should be aimed. The influence of parameters on these conflicting requirements was examined in order to derive design-related technological recommendations. Due to adjusted process parameters, results representing a compromise between the competing application criteria can be achieved. Apertures that have been manufactured with the described method, are used in Nanosatellites as Technosat [7], for example. In order to measure the influences during the individual processing steps different sample geometries were used. The sample geometries include cylindrical and triangular raised structures and optical apertures with a starting diameter of 300 µm (Figure 1). Each set of test geometries has been milled into one individual test sample and was measured and processed afterwards. Figure 2 shows the typical errors that occur during the process. The consequences of long anodizing times are significant deviations from the nominal value and brittle fractures at the structure edges. On the other hand, short anodizing times cause high, remaining reflectivity.

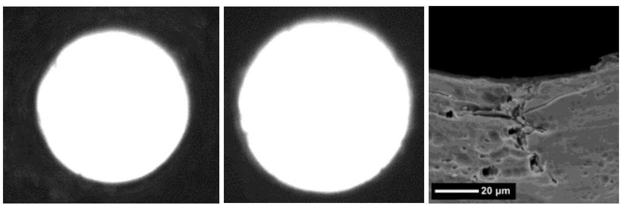

Figure 2. Exemplary errors (left: ok; middle: too large; right: SEM crack)

2. Influence of process parameters

The experimental setup consists of a galvanic cell and the anodic workpiece, located between two lead cathodes. By means of a laboratory power supply the current (and thus the current density) is regulated with an accuracy of ΔI = 0,01 A. The coating process is a sequence of etching in 10 % NaOH, the anodizing step in 17 % H_2SO_4 and a colouration process. Dimensional losses occur in particular in the first two steps. The variable parameters are etching-, anodizing time and current density. The experiments are a combination of DOE and OFAT.

2.1. Influence on the structure dimensions

The measurement of the structures was carried out on the ultraprecision machine by a measuring camera with a resolution of 0,5 µm per pixel. The analysis was carried out automatically by means of edge detection.

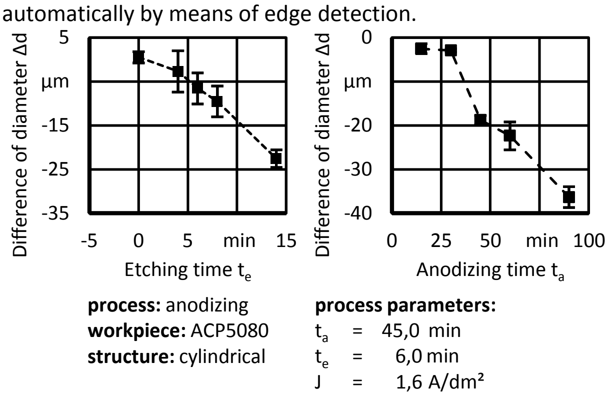

Figure 3. Influence of etching- and anodizing time on the accuracy

Considering the etching time (OFAT), the expected linear removal occurs for a regression of 1,7 µm/min. Considering the anodizing time, one realizes that initially only a slight removal occurs, which considerably increases suddenly to a linear removal with a regression of 0,55 µm/min. (Figure 3). This can be explained by the simultaneous material removal and layer growth. The total thickness of the oxide layer reaches its upper limit when growth and removal are balanced. If this condition of equilibrium is reached, a close to linear erosion of the material occurs. With regard to a high structural accuracy, the process must be stopped before reaching the condition of equilibrium. With respect to the reflectivity, it is advisable to use the exact process point as process stop, hence the layer is not too thin.

2.2. Influence on the reflectivity

The reflectivity was measured with a photospectrometre Konica Minolta CM-700d. The measurements were carried out in a wavelength range of 400 nm < λ < 700 nm. Figure 4 shows the typical curve of exemplary measurements. It is advantageous that the reflectivity is reduced over the entire wavelength range, especially for wavelengths longer than λ = 650 nm, which results in an increased overall reflectivity.
In order to classify the results absolutely, colour measurements in the Lab colour space were performed supplementary and compared to an industrial reference.

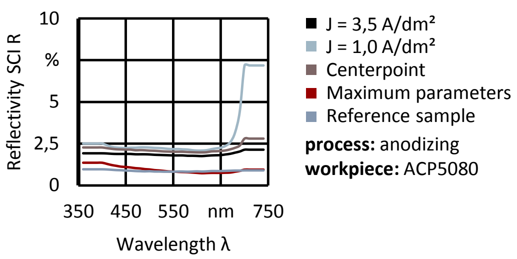

Figure 4. Sample curves of reflectivity due to different parameter sets

The measurements were carried out based on a D65 illumination norm spectrum (DIN 6173). The reference was measured in an identical manner as the experimental samples and is classified in Lab space with the values L* = 7,41 | a* = - 0,49 | b* = - 0,99. The values in the experimental series shift between L* = 22,55 | a* = - 0,52 | b* = - 3,16 and L* = 8,59 | a* = 0,18 | b* = - 1,93 and are very close to the reference value in their maximum blackening.

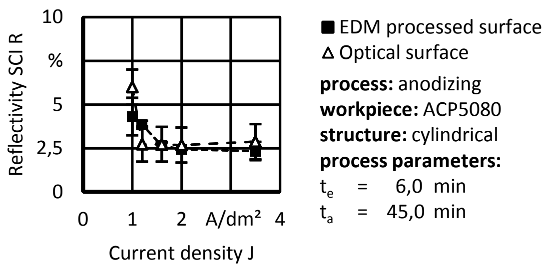

Figure 5. Influence of current density on the mean reflectivity

The most significant impact on the colour and mean reflectivity is represented by the current density, what can be explained by an increasing layer thickness [3]. Figure 5 shows the curve of reflectivity for two different starting surfaces. Both behave asymptotically almost identical and reach minimum at about J = 1,6 A/dm².

3. Summary and discussion

It could be shown that the anodizing process is a suitable method to reduce reflection in micro optical applications. It can be applied with a low tolerance on microstructures. Moreover, it was shown that the current density is most critical for the reflectivity. As expected the etching time has a high impact on the structural quality, but in spite of the strong surface alteration, only a small influence on the reflectivity.
Due to process parameters, representing a compromise between competing application criteria (reflectance and dimensional accuracy), an outstanding result was achieved with a reflectivity of R = 3,3 % and a dimensional accuracy of 3 µm. In spite of the compromise parameters the Lab values of the sample are with L* = 15,4 | a* = 0,25 | b* = 0,13 near those of the industrial reference sample.

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