

Through-Mask Electrochemical Micromachining of Titanium Applied to Biological Model Surfaces

O. Zinger and D. Landolt

Laboratoire de Métallurgie Chimique, Département des Matériaux, Ecole Polytechnique Fédérale de Lausanne, CH-1015 Lausanne EPFL, Switzerland

Introduction

Biological performances of implantable titanium devices in medicine and dentistry have been shown to depend on their surface topography in the micro and nanometer range [1]. Thus, one needs well-defined model surfaces to achieve a better understanding of the effect of surface topography on cell adhesion and differentiation.

The chemical stability of titanium results from the presence of a thin passive film of a few nanometers. In nonaqueous electrolytes containing perchloric or sulfuric acid, the oxide film is unstable and anodic polarization leads to titanium dissolution at high rate. Recently, a sulfuric acid based-methanol electrolyte has been developed for electrochemical polishing of titanium [2]. Best polishing was obtained in an electrolyte containing 3 M sulfuric acid for applied potentials above 8 V corresponding to mass-transport controlled dissolution conditions. Using this electrolyte, well-defined topographies in the micrometer range were produced on bulk titanium by electrochemical micromachining through a patterned photoresist [3,4].

The aim of this paper is to develop electrochemical procedures that lead to improved reproducibility and more precise shape control in through-mask electrochemical micromachining of titanium. For this, a new holder providing in-situ sample cooling was designed and the electrochemical parameters were optimized. Application of a potential step, followed by a linear potential decrease, allowed for efficient removal of the passive film and anodic metal dissolution without excessive Joule heating.

Experimental

Resist patterning by photolithography

Commercially pure titanium disks (Ti 99.6%) were mechanically polished to obtain a mirror finish surface. The polished titanium was coated with a negative polyimide based photoresist, which was exposed using a standard UV mask aligner and developed to reveal the initial patterns.

Electrochemical dissolution of titanium

The dissolution of the titanium through the patterned photoresist was performed in a methanol based electropolishing electrolyte with 3 M sulfuric acid [4].

Results

High dissolution rates can lead to Joule heating, affecting precision of the attack and therefore reproducibility. Joule heating is important for a pattern with a large etched surface area and/or high density. To reduce this effect, the electrolyte is cooled down to -10°C ; ethanol was added to the methanol (50/50 mixture) to increase viscosity and decrease the anodic current.

To study the role of topography on the interactions of cells with implants, specific patterns were designed; some of them having a very high density, still resulting in excessive Joule heating. Fig. 1 shows a hole presenting a bad surface finish coming from an excess of heat. To be able to dissolve titanium with smooth surface finish and good reproducibility, a special sample-holder cooled from the inside was developed, and electrochemical parameters were optimized. First, a short polarization of 40 V was

applied to remove the titanium oxide as quick as possible to obtain an homogeneous dissolution from the beginning of the process; then, to reduce Joule heating effects, the potential was linearly decreased to 15 V and maintained at this voltage until the end of the dissolution. Fig. 2 presents a 30 μm diameter hole with smooth surface texture and sharp edge obtained under these optimized conditions. Fig. 3 shows an array of the same holes in hexagonal compact arrangement; the separation is 3 μm .

Conclusion

The optimization of electrochemical parameters and the use of a cooled sample-holder enabled us to produce dense and well-defined surface structures in the micrometer range on bulk titanium with smooth surface finish and good reproducibility. These structures will serve as model surfaces to study the role of the surface topography on the interactions of cells with implants.

References

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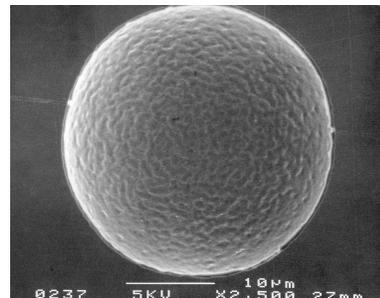


Fig. 1 SEM micrograph of a hole (\varnothing 30 μm) dissolved with non-optimized electrochemical parameters

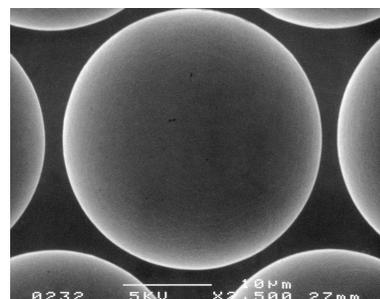


Fig. 2 SEM micrograph of a hole (\varnothing 30 μm) dissolved with optimized electrochemical parameters

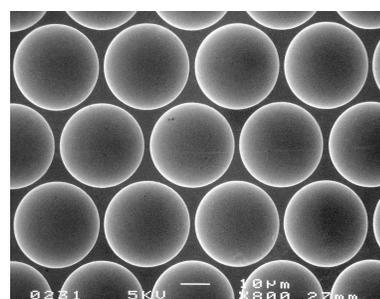


Fig. 3 SEM micrograph of an array of holes (\varnothing 30 μm) in hexagonal compact arrangement