Microelectronic Engineering 164 (2016) 36–42

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Research paper

Fabrication of mesoscale topographical gradients in bulk titanium and their use in injection moulding

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ARTICLE INFO

Article history:
Received 17 March 2016
Received in revised form 11 July 2016
Accepted 13 July 2016
Available online 15 July 2016

Keywords:
Gradients
Titanium etching
Plasma polymerisation
Injection moulding

ABSTRACT

Fabrication methods for titanium substrates exhibiting continuous micro and nano scale arrays, with increasing feature heights over the length of the array are reported. The resultant feature heights spanned 0–2 μm. Patterned gradient arrays of circular features with diameters of: 500 nm, 1 μm and 2 μm, spaced by twice the diameter were manufactured by the process using specially prepared titanium substrates. Patterns were exposed by electron beam lithography and the length of the patterned arrays was 15 mm or 20 mm. This work presents two selectivity amplification processes to achieve a gradient of feature heights ranging over the titanium array after consecutive reactive ion etching processes. The first, route A: a HSQ on Ti, gradient amplification process. The second, route B, a SiO_2 layer amplification transfer into Ti. The crucial initial gradient component deposited for the amplification process for both routes was a diffusion limited plasma polymerised hexane gradient. Etching using respective reactive ion etch chemistries for each gradient transfer through the various selectivity amplification layers (employing consecutive etch steps, in this way) enables a dual amplification for each route to manufacture. The original gradient is transferred into titanium as a function of the sum of the respective selectivities between the materials, using the appropriate dry etch plasma conditions. The substrates henceforth are referred to as inlays, and were tested for use as a high throughput platform for polymer replication by injection moulding. It is envisaged that the fabrication methodology and resultant topographies have use in a range of engineering applications. The overall selectivity to Ti for polymerised hexane is increased by more than 20 times using each dual amplification process.

1. Introduction

Bio-orientated gradient topographies are presented here. This research motivation proceeds from work which in the past has employed gels, discrete pillar topographies and limited height range pillar gradient topographies to establish that cells are influenced in many ways via a mechanism called mechanotransduction [1–4]. As the name suggests, this mechanotransductive property cells exhibit, refers to the process by which a cell transduces a force into a biological response [3,4]. Implicitly, it has been shown that the associated tension of a cell's cytoskeleton can be influenced by the stiffness of a culture substrate. The resultant forces of interaction on or by the cell, in turn affects the mechanotransductive processes. This was elucidated by varying the stiffness's of the culture matrices, which can influence the cell's phenotype, proteomic expression, biochemical signalling activity and overall homeostasis [2,4–6]. The understanding of this synergistic cell-substrate mechanism is, however, in its infancy [1,6].

Feature gradient substrates for polymer injection moulding or "inlays" and their polymer replicates which can be later used as moulds to cast elastomeric polymers from, have been manufactured. This is specifically to provide for a prospective cell culture substrate with an associated pillar stiffness spectrum spanning the known range of cellular influence [3,5,7]. Diffusion limited plasma polymerised hexane gradients (ppHex) have been crucially utilised to provide part of this novel gradient amplification manufacture process [8,9]. Titanium (grade II) was used as the bulk material into which the gradient amplification was made, via one the two individual selectivity amplification mediums trialled. The amplification was enabled via ppHex gradient profile transfer through the sacrificial layer by reactive ion etching (RIE). Although traditionally considered difficult, titanium tooling was used as the bulk material for fabrication of this high aspect ratio (HAR) inlay for polymer injection moulding [9,10]. Better inlay fill and a slower rate of polymer cooling within or around the features of a titanium inlay versus some other tooling solutions is exhibited [9]. The thermal performance of titanium also negates the need for variothermal heat retardation techniques to achieve amiable replication. Such complicated and expensive techniques involve plumbing or electrical heating at the inlay tool interface of the injection moulder to control and improve inlay fill,

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http://dx.doi.org/10.1016/j.mee.2016.07.004

polymer freezing and help optimise cycle time [9]. Other additions to the inlay fabrication process like surface modification by silanization or plasma treatment techniques are not necessary using this titanium fabrication methodology. Titanium also exhibits better corrosion resistance and stress tolerance than most of all other materials commonly used for polymer injection moulding tooling today.

2. Materials and methods

2.1. Titanium polishing

Water jet cut titanium pieces of 25 mm × 25 mm × 1 mm with an unprocessed surface roughness of approximately 3.5 μm Ra were polished by hand polishing and chemical mechanical polishing (CMP). Hand polishing was initially performed by sanding the titanium surface with sandpaper consecutively using 200, 450, and then 600 grade grit. Following this, felt brushes and a hand drill were used to polish the Ti surface using diamond polishing paste of 2 μm and 1 μm particle size. Next, an automated chemical mechanical polishing machine (Orbis, CMP) was used with nanometre scale SiO_2 grit (slurry, ~30 nm) to achieve a mirror finish. After polishing, the titanium substrates were sonicated in MF-319 microposit developer for 5 min. This assists removal of the contaminants from the hand polishing stage and also silica embedded in the surface within the grain boundaries of the Ti after CMP processing. Samples were subsequently cleaned by sonication in acetone, methanol, and IPA respectively for 5 min each. Averaged AFM microscopy shows a polishing capability of 3.0 nm (±1 nm) Ra. The average deviation in surface roughness was calculated by measurements taken over 3 samples, with 6 measurements each of scan size 12 × 4 μm on each sample scan (NanoScope software).

2.2. Sample preparation

Samples for route A, (Fig. 1A) were prepared for electron beam exposure by spinning a pipetted volume of 250 μl HSQ (Hydrogen silsesquioxane, Dow Corning) onto the titanium substrate at 6000 rpm for 60 s. This yielded an approximate layer thickness of 310 nm. The substrates were baked for 2.5 min at 90 °C on a hotplate. Post electron beam exposure, HSQ substrates were developed at ambient temperature (20 °C), using CD-26 for 30 s. An IPA wash bottle rinse and subsequent DI water rinse (for 2 & 5 min respectively) post development was found to reduce micro masking effects after the subsequent etching. For substrates which utilised a SiO_2 layer i.e. route B, (Fig. 1B), the deposition was performed by plasma enhanced chemical vapour deposition (PEVCD 80 Plus, Oxford Instruments). For route B, 8% conc. 2010 PMMA (Elvacite 2010, Lucite International) was spun onto a substrate at 4000 rpm for 60 s. Then a bilayer was spun by spinning a second resist layer atop the primary PMMA layer using 4% conc. 2041 PMMA for 60 s, at 5000 rpm (Elvacite 2041, Lucite International). The overall thickness was ~410 nm. This bi-layer later serves to provide masking for the deposition of a patterned nichrome (NiCr) metal lift-off layer (65 nm, Plassys MEB 550S, electron beam evaporator), after exposure and development of the resist (see Fig. 1B). After spinning, the substrate was oven baked at 120 °C for 15 min then transferred to a 180 °C oven for 8 h. A conductance layer of 30 nm aluminium was then evaporated onto the substrate.

2.3. Electron beam lithography

The thermal field emission gun was 100 keV (as standard for Vistec, VB-6), a 32 nA beam current was selected. The beam step size chosen was 19 nm and was used for the exposure of both resists for this

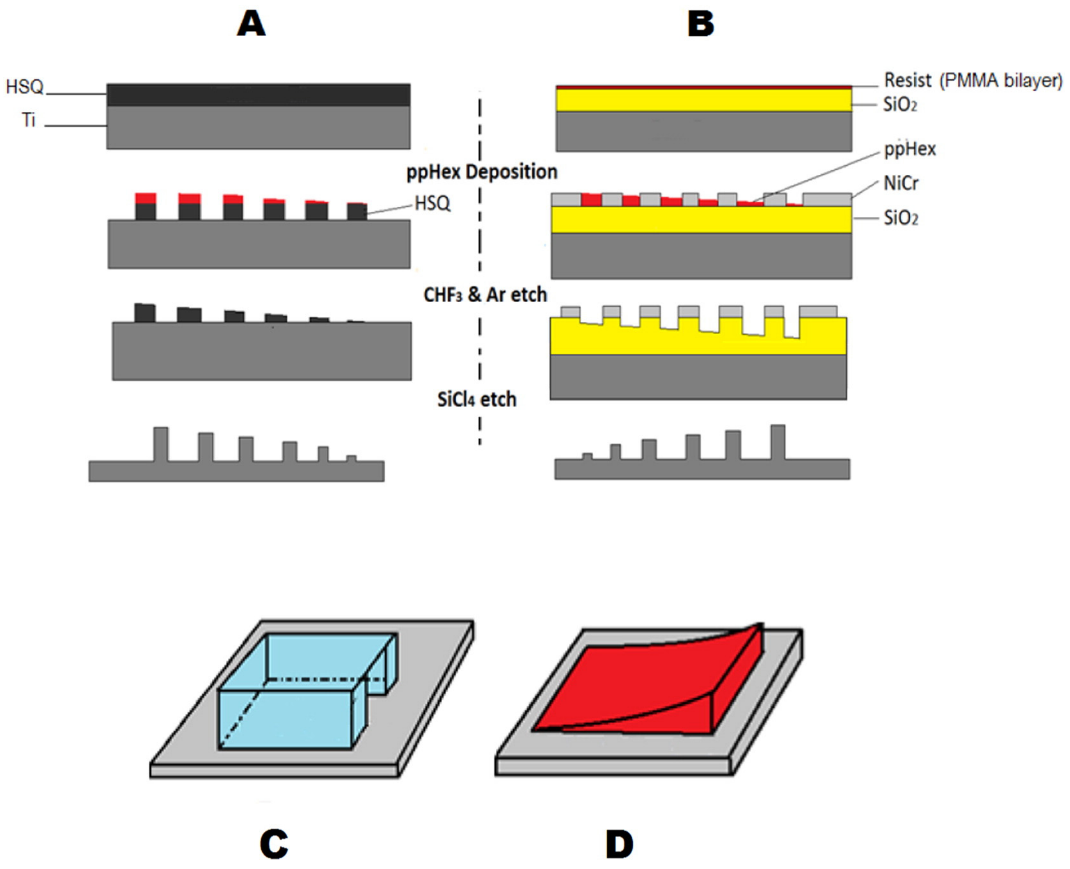

Fig. 1. (A) and (B) depict two routes to manufacture for the fabrication of pillar gradient arrays, made of titanium. (C) A diffusion limiting mask of dimensions: 20 × 15 × 8 mm, (D) a schematic of a ppHex gradient profile (mask removed) on a piece of titanium post deposition. Figure is not to scale.