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Nickel electroplating for high-resolution nanostructures

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ABSTRACT

We present optimisation of a nickel electroplating process in order to obtain high-resolution nanostructures. Film stress and roughness were minimised and the deposition rate was well controlled at ~36 nm/min with optimised parameters of current density, pulse period, and duty cycle. The thickness uniformity of the film was measured to be within 5% across the 9 mm^2 sample with an R.M.S. roughness of ~4 nm. We further demonstrate electro-deposition of nano-sized lines and pillars using these parameters. Structures showed good resolution and uniformity down to 20 nm while pillars of 70 nm diameter with aspect ratios up to 1:10 were fabricated. This process is currently being used in the fabrication of high quality EUV interference lithography transmission masks in our group, and could be used in future research and applications such as wire grid polarisers, Fresnel zone plates, and plasmonics.

1. Introduction

Presently, the applications of thin-film nickel (Ni) layers are wide-ranging. The electroplating process is an effective method for obtaining Ni thin films or micro- and nanostructures with good film quality and aspect ratio. Reported applications include fabrication of durable moulds for nano-imprint lithography [1] and the alteration of a compound's magnetic properties for Ultra-Large-Scale Integration (ULSI) technology products, such as high-density recording systems [2]. Nickel films are increasingly being used in the fabrication of lithography masks [3,4]. Ni is also an attractive material for diffractive optical elements for soft X-rays. In this energy range, Ni provides a phase shift and thereby enables fabrication of efficient diffractive optical elements such as gratings and Fresnel zone plates [5–7]. Fresnel zone plates with 20 nm outer zone width, although rough, have been shown [8]. For these applications, not only smoothness of Ni, but also its stress is decisive, since these optical elements are fabricated on thin membranes which cannot withstand high mechanical stress.

The specific aim of this study was to optimise the Ni deposition process in order to use Ni gratings for interference lithography at the wavelength range of extreme ultra violet (EUV). In interference lithography the interference pattern is obtained from the diffracted beams of gratings [9,10]. Since the interference pattern is formed between the gratings, the incident light between the gratings must be effectively blocked. Typically, this is done with a 400 nm thick Au film [9]. However, Ni has an attenuation length of roughly two-thirds that of Au at the EUV wavelength of 13.5 nm [11,12]. Therefore, in order to obtain the same level of attenuation, a thinner layer of Ni would be required. Consequently, the mask on which the Ni layer is being deposited would experience less stress, resulting in higher mask durability. Moreover, Ni gratings can be used as an effective grating with relatively low thicknesses due to their short attenuation length at EUV wavelength.

In order for this Ni layer to perform satisfactorily, its quality, as well as that of its fabrication procedure, must be ensured. The parameters that were identified to be the most essential were uniformity of deposit and film stress. A lack of the former would result in a poor and inconsistent fabrication process, while if the stress experienced is too large, it would lead to mask failure. The final parameter that needs to be analysed in depth is the deposition rate. Knowledge of the Ni growth rate is essential in order to be able to accurately and reproducibly deposit the desired metal thickness. Here we report on an extensive optimisation of Ni electroplating which provides fabrication of Ni nanostructures with low roughness and stress and high resolution. These results enable realisation of diffractive optical elements for EUV and soft X-ray ranges as well as Ni nanostructures for various applications with increased resolution, quality, and stability.

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2. Methodology and results

2.1. Experimental setup and sample preparation

Various Ni deposition techniques have been documented, with the most common being electroplating [14], electroforming [1] and electroless-plating [15]. In an electroplating setup, such as the one shown in Fig. 1(a), a current is passed from an anode to a cathode through an electrolyte causing the dissociated metal ions to gain electrons and oxidise on the surface of the cathode, thereby forming a metallic coating. Electroforming is most useful when the deposit needs to be separated from the cathode base. On the other hand, electroless-plating tends to result in the deposition of a compound which, in the case of Ni, is usually between 90% and 98% Ni. In contrast, electroplating always deposits over 99% Ni [16] and allows for greater control over the process since it depends on the current forced through the electrolyte. Owing to these advantages, electroplating is mostly preferred to other Ni deposition techniques.

Previous work on nickel electroplating showed that nickel deposition rate varies for different locations on the sample, which can be controlled through use of pulsed and pulse reverse plating [7]. Luo et al. [13] performed a detailed study of thin film nickel electroplating, showing the relationship between stress and temperature, with an optimal temperature of ~60 °C. This led to the conclusion that temperature control and relaxation through current pulsing was essential for low stress, and an optimal current density of 2 mA/cm^2 gave stress-free thin films.

As previously mentioned, the electrical current input provides control over the process. In the present study, we used a power supply (Plating Electronic, PE86CB) which is capable of producing stable unipolar rectangular pulses through a control feedback loop. In this study we investigate all three of the current waveform's variable parameters, namely the current amplitude, the duty cycle, and the pulse time (i.e., waveform period).

In order to obtain reliable and repeatable results, the electroplating bath's operating conditions had to be monitored and maintained as stable and as consistent as possible. The operating conditions of the nickel sulphamate electrolyte solution (185 g/L concentration, pH 3.0, 57 °C) are in line with those recommended by the supplier (Enthone Inc.). The magnetic stirrer, as seen in Fig. 1(a), serves to maintain an even ion distribution throughout the whole bath, thereby maximising deposit uniformity.

Apart from the current waveform and the bath operating conditions, the process is also dependent on the cathode's conductive area. For this reason, an 18 × 30 mm dummy Si wafer was introduced. In this way, changes in the sample's area are negligible and the current density is kept constant for different samples. The samples consisted of Si chips and 100 nm thick Si_3N_4 membranes with a 25 nm Au seed layer on top of a 5 nm Cr adhesion layer [17]. The Si dummy was coated with a 20 nm Au seed layer. The patterned Cr/Au bilayer was fabricated by spin-coating PMMA 950k (4% wt. in Ethyllactate, AR-P 679.04) at 2500 rpm, followed by post application bake, e-beam exposure, development, metal evaporation, and lift-off in acetone to remove the PMMA. For fabrication of nickel nanostructures, the PMMA was applied

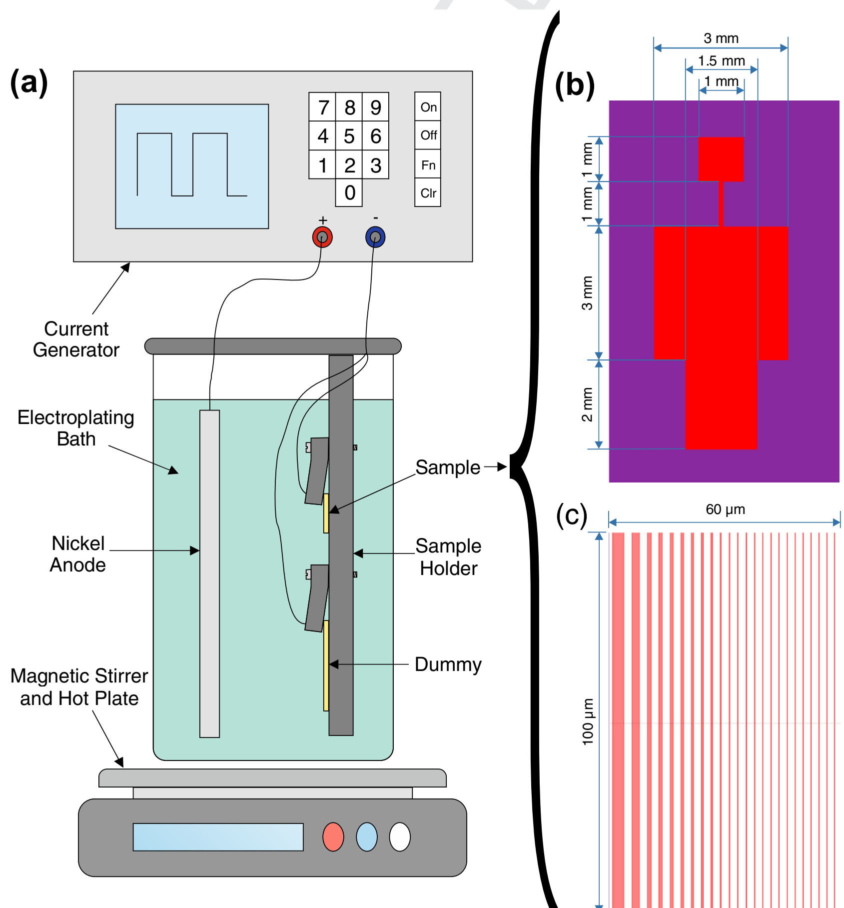

Fig. 1. (a) Schematic of the electroplating setup. (b) Testing sample design used for characterisation purposes. (c) Testing sample design used for fabrication of a line pattern with variable widths.

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