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Sub-10 nm Electron and Helium Ion Beam Lithography Using a Recently Developed Alumina Resist

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Abstract

Electron beam lithography (EBL) at sub-10 nm resolution is mainly limited by resist contrast and proximity effects. In this work, we investigate the use of a recently developed alumina-based resist as a negative-tone resist for electron EBL at 100 keV and focused helium ion beam lithography (FHIBL). The resist is synthesized using a sol-gel method and turns into a near completely inorganic alumina system when exposed to the electron/ion beam. We first investigate the effect on the resist contrast curve on i) development temperature; ii) stability of the resist after exposure and before post-baking and development; and iii) aging of the resist solution. We demonstrate the patterning of isolated features as small as 6.5 nm using an EBL and 5 nm using FHIBL and a resolution down to 10 nm for FHIB exposed films. Finally, we demonstrate the pattern transfer of 10 nm lines with an aspect ratio of 10 in silicon, using an optimized reactive ion etching process.

1. Introduction

Electron Beam Lithography (EBL) at sub-10 nm resolution is mainly limited by resist contrast and the point-spread function of the exposing beam. In the case of high-resolution silsesquioxane (HSQ) negative-tone resist, much work was devoted to improving the contrast of the development process using hot development,[1,2,3,4] KOH development,[5,6] or salty development.[7] Some authors have addressed the problem using focused helium-ion beam lithography (FHIBL), which features smaller proximity effects as compared to an electron beam at the same incident kinetic energy.[8,9,10] In addition to improved resolution and line edge roughness, the development of new EBL resists has also been triggered by the need of high sensitivities resists[11] and environmental benign development processes.[12] Despite the high resolution demonstrated using these approaches, the problem of pattern transfer is rarely addressed and remains problematic a this scale, mainly due to the relatively low plasma etching resistance of HSQ or polymeric resists for fluorinated plasmas used in silicon dry etching.

Recently, the group of Brusantin developed a hybrid organic-inorganic alumina-based resist for electron- or photon-based nanolithography.[13] The resist is synthesized using a sol-gel method and turns into an almost completely inorganic alumina system when exposed to the electron beam. The modification of the organic component of the exposed areas leads to the possibility of developing the resist as a negative-tone resist dissolving the organic unexposed part in hydrochloric acid (HCl) : isopropyl alcohol (IPA) solution or as a positive-tone resist dissolving the inorganic exposed part in buffered oxide etch (BOE). Used as negative-tone resist for EBL (at 3 keV) the authors of the study demonstrated isolated features down to 11 nm and an exceptional selectivity (100:1) when used as a mask for inductively coupled plasma (ICP) etching of silicon in fluorinated plasmas.[14]

In this work - performed within the framework of the EU project NFFA-Europe (Nanoscience Foundries and Fine Analysis), the H2020 framework program for research and innovation, and grant agreement 654360 - we explore the possibility to use such an alumina-based resist for sub-10 nm patterning of silicon. The resist is used as a negative-tone resist and exposed using an EBL system at 100 keV as well as a FHIBL system at 30 keV. Pattern transfer in silicon was performed using a reactive ion etching (RIE) with a relatively low bias in order to improve the selectivity. The results of this work will be used in the Transnational Access program within the NFFA-Europe project to provide access to ultra-high resolution patterning techniques.

2. Materials and Methods

The resist is synthesized using a sol-gel method starting with aluminum-tri-sec-butoxide (97%, Aldrich) and triethoxyphenylsilane (98%, Aldrich). Note that we replaced the trimethoxyphenylsilane used in the original publications[13,14] with the less toxic triethoxyphenylsilane. The aluminum-tri-sec-butoxide was left stirring 1 h at 70 °C in a solution of methoxyethanol and acetic acid in the molar ratio of 1:21:4.3. After 1 h the triethoxyphenylsilane was added in a molar ratio of 0.25 and the solution stirred for another hour at 70 °C.

Silicon substrates were spin-coated at 5000 rpm with the sol diluted in methoxyethanol with variable dilution to give adjusted film thicknesses of 35 and 20 nm. The samples were pre-baked for 2 min at 100 °C in order to remove