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Thin Solid Films 504 (2006) 113 – 116
thin solid films
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Characterization of chemically amplified resist for X-ray lithography by Fourier transform infrared spectroscopy

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Available online 20 October 2005

Abstract

SU-8 resist was characterized for X-ray lithography from a plasma focus source by studying its cross-linking process using Fourier transform infrared (FT-IR) spectroscopy. The cross-linking process of the resist during post-exposure bake (PEB) was accurately monitored using the infrared absorption peaks at 862, 914, and 1128 cm^{-1}. Results showed that the cross-linking of SU-8 was effectively completed at the exposure dose of 2500 mJ/cm^2 for resist thickness of 25 μm. Reliable processing conditions consisted of an intermediate PEB at 65 °C for 5 min, with the PEB temperature ramped up to 95 °C over 1.5 min and then followed by a final PEB at 95 °C for 5 min. Test structures with aspect ratio 20:1 were obtained.

Keywords: Chemically amplified resist SU-8

1. Introduction

Various lithographic studies on SU-8 have been performed using a variety of radiation sources—ultraviolet [1–3], X-rays [4–7], proton beam [8] and electron beam [9–11]. In particular, the short wavelength and high penetration power of X-rays allow microstructures with submicron resolution and high aspect ratio to be produced. A previous study [12] on the characterization of SU-8 for X-ray Lithography (XRL) using the dense plasma focus (DPF) device was made on resist thickness of 3–15 μm. In this investigation, the processing parameters of the 25-μm-thick SU-8 resist for X-ray lithographic applications are derived.

2. Experimental details

For our study, we used the SU-8 2010 formulation with a viscosity of 1050 cst (at 25 °C), obtained commercially from MicroChem Corporation. The SU-8 resist consists of a multifunctional, highly branched polymeric epoxy resin dissolved in an organic solvent, gamma-butyrolactone (GBL). Along with the formulation is a triaryl sulfonium salt which acts as a photoacid generator. The resist has on average 8 epoxy ring groups in each molecule; hence the name 'SU-8'. Its chemical structure is shown in Fig. 1.

2.1. Thickness measurements of resist layer

The SU-8 resist was spin-coated at various speeds of 500–2500 rpm. Care was taken to remove any excess resist from the edge and bottom of the substrate. The resist film was then subjected to a 15-min soft-bake at 95 °C on a level hot plate in order to remove the solvent from the resist. It was found that the spin speed of 500 rpm gave about 25-μm-thick samples. The thickness of the resist was measured using the oscillating patterns along the baseline of the FT-IR spectrum [12], as shown from Fig. 2.

2.2. Resist exposure

The 25 μm resist samples were irradiated by soft X-rays (SXRs) emitted from a DPF device. Such a device operates with a pulsed capacitive discharge, where a dense, magnetically compressed plasma is produced at the end of the two coaxial electrodes. This is followed by the decay of the plasma column, giving rise to X-rays, amongst a host of other radiation. The details of the set-up of the DPF device are found elsewhere [13].

A 10-μm Be filter was used to shield off visible and ultraviolet light from irradiating the samples. Based on the X-ray

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doi:10.1016/j.tsf.2005.09.151

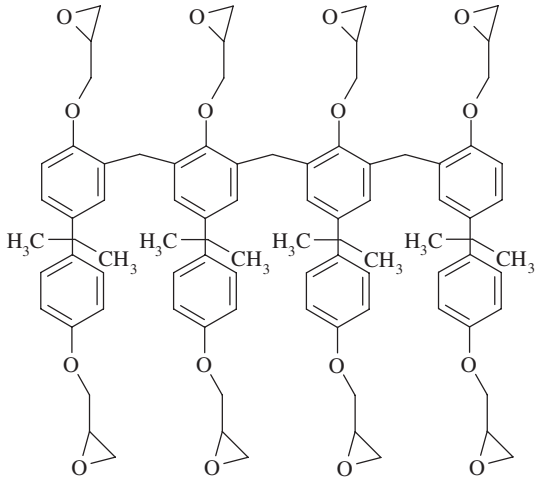

Fig. 1. Chemical structure of SU-8.

spectral distribution given off by the DPF device, the average weighted energy is found to be about 1.0 keV. However, because of the coupled effect of the Be filter and Ne gas transmission characteristics, the average weighted energy falling on the resist is actually slightly higher.

Previous experiments [12] showed that for a resist of thickness 10 μm, a dosage of about 250 mJ/cm^2 was sufficient for structures to be formed, although with slightly incomplete cross-linking at the bottom surface. The estimated X-ray transmission curves of SU-8 (density = 1.2 g/cm^3) for resist thickness of 10, 15 and 25 μm are shown in Fig. 3. For X-ray energy of 1.1 keV, the transmission after passing through 10 μm of SU-8 resist is about 0.1. Hence we estimate that to maintain the same minimum exposure dose at the bottom surface for a 25-μm resist, we need about ten-fold an amount of X-rays to be irradiated at the top surface (2500 mJ/cm^2).

3. Results and discussion

3.1. Degree of cross-linking

The FT-IR spectrum for pre-exposed SU-8 is given in Fig. 4. The infrared absorption peaks of the epoxide ring modes at 914 and 862 cm^{-1} were observed to gradually reduce in intensity

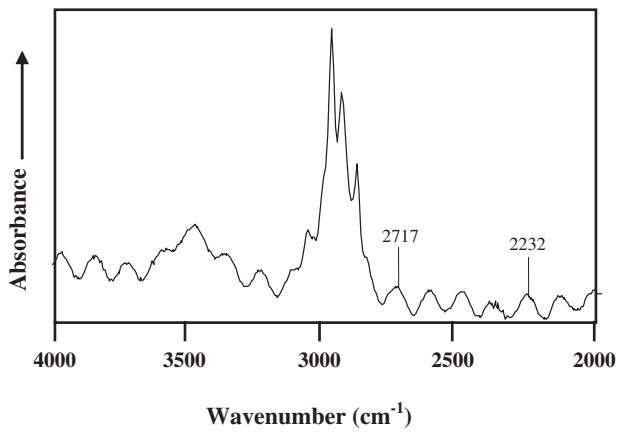

Fig. 2. FT-IR spectra of SU-8 resist at spin speed of 500 rpm.

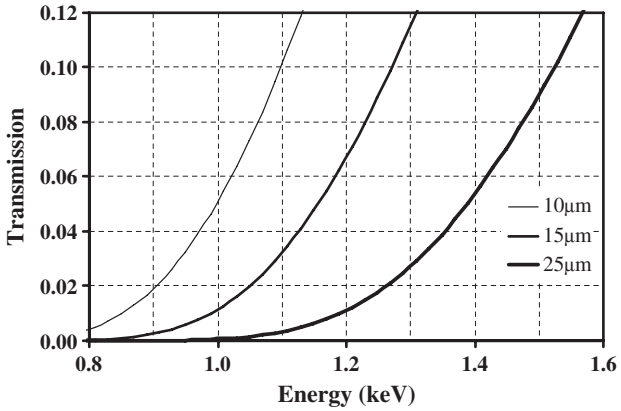

Fig. 3. Estimated X-ray transmission curves of SU-8 (density = 1.2 g/cm^3) for resist thickness of 10, 15 and 25 μm.

with increased exposure dose after PEB. Conversely, intensity of the 1128 cm^{-1} absorption peak associated with the ether bond gradually increased. The epoxide peaks at 914 and 862 cm^{-1} were selected for quantitative determination of the degree of cross-linking.

In an effort to eliminate errors due to uneven thickness, an infrared peak, which does not participate in the cross-linking process is normally used as an internal standard, and all other peaks are normalized against this peak. The aromatic ring C–C stretch mode at 1608 cm^{-1} was selected for this purpose. The intensity of the peak at 1608 cm^{-1} is comparable to that at 914 cm^{-1} and hence the value of the normalized intensity given as the ratio (I_{914}/I_{1608}) gives the most reliable measure for the degree of cross-linking.

3.2. Post-exposure bake

The samples were subjected to varying PEB temperatures of 65–120 °C in order to initiate the cross-linking process. After PEB, FT-IR spectra of the resist samples were recorded. To prevent resist cracking on large exposed areas, we applied an intermediate bake at 65 °C for 5 min for each sample. This was followed by ramping at the rate of about 20 °C/min and with the samples subjected to the desired temperature for another 5

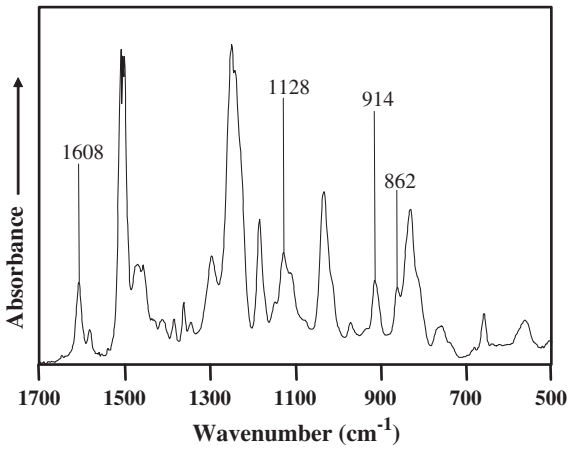

Fig. 4. FT-IR spectra of non-cross-linked SU-8.