In-situ Raman spectroscopy characterization of SU-8 epoxy resin temperature dependent curing process

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Abstract. *The SU-8 is commonly used epoxy-based photoresist in manufacturing microfluidics and microelectromechanical systems (MEMS). In this paper, we investigated the temperature dependent polymerization process of SU-8 using Raman spectroscopy. The Raman spectrum were recorded in-situ during the heating of the sample from 25 to 160 degrees of Celsius. From the collected spectrum we can observe an increase in the intensity of 1183 cm^{-1} and 1108 cm^{-1} peaks corresponding to the asymmetric vibrations of C-O-C bond in polymer and decrease in the intensity of 930 cm^{-1} peaks attributed to symmetric vibration of C-O-C bond in the polymer.*

Keywords

Raman spectroscopy, SU-8, polymerization

1. Introduction

In this introduction study, we examined the changes in SU-8 photoresists by Raman spectroscopy during thermal curing. The SU-8 is a high contract epoxy-based negative photoresist with a high aspect ratio designed for application with a necessity for chemically and thermally stable films or structures. As the name suggests, it contains eight epoxy groups. The Su-8 was originally developed as a photoresist used in microelectronics for creating high-resolution masks, but recently it is used mainly in manufacturing microfluidics and microelectromechanical systems. For that application, a controlled hard-bake process is recommended to further cross-link the SU-8 structures.

The Raman effect is called after sir C.V.Raman, who first successfully demonstrated inelastic scattering of light by fluid in 1928. In Raman spectrometer, the sample is irradiated by an intense source of monochromatic light, for which he used filtered sunlight. He was awarded Nobel prize in physics in 1930 [1]. The Raman scattering can be described as an inelastic collision between an incident photon and the molecule, which result in change of molecules vibrational or rotational energy. In order for the necessity of energy conservation, the energy of the scattered photon must be different from the energy of incident photon [2]. This can be referred to as the Raman shift and is usually displayed in cm^{-1}. Various studies involving Raman characterization of SU-8 during different polymerization conditions were performed. The changes in the Raman scattering of SU-8 photoresist were studied by Suzuki et al [3].

2. Experimental

The SU-8 2 epoxy-based resin was obtained from MicroChem Inc. It was drop cast onto a glass slide with thermally evaporated 50 nm thin alumina layer for better reflexivity of the substrate. The sample was then soft-baked for 20 minutes at 60 °C at the hot plate. The Raman spectrum of the samples was collected by Renishaw inVia™ Qontor ® confocal Raman spectrometer. As the excitation source, we used 830 nm laser. After the soft-bake, the sample was placed inside the LINKAM chamber, equipped with an embedded heater, which was placed in the Raman spectrometer. The measurement setup can be seen in Figure 1.

Fig. 1. The LINKAM chamber with embedded heater inside the Raman spectrometer.

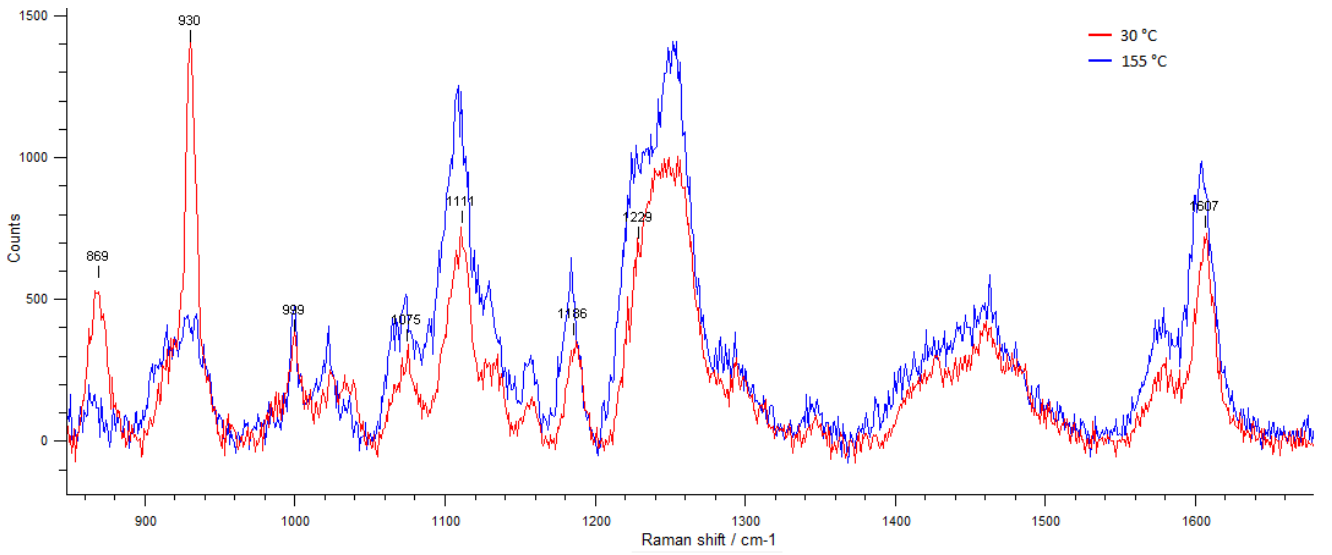

Fig. 2. The Raman spectra at the 30°C in red and in blue at the end of the hard-bake curing process at 155°C.

The Raman spectrum, we obtained in-situ during the curing process. It consisted of heat ramp from 25 to 120 °C at the rate of 5 °C per minute, then the temperature was kept at 120 °C for 20 minutes and finally ramped up to 160 °C at the same rate of 5°C per minute. The Raman spectrum was collected every minute during the heat ramp up, collecting first spectra at 30 °C. During the 120 °C period, we collected the spectrum every two minutes and every minute at the final ramp up, collecting last spectra at the temperature of 155 °C.

3. Results and discussion

In Figure 2., we can see the spectrum of the SU-8 at 30 °C in the red color and in the blue color the last spectra from the data set, collected at 155 °C. From the obtained Raman spectra we can observe the disappearance of the 869 peaks which can be attributed to the evaporation of the solvent. There is a strong decrease in the peak of 930 cm^{-1} associated to the symmetric stretching vibration of C-O-C bond and increase in the intensity of the peak around 1108 cm^{-1} and 1180 cm^{-1} which is associated asymmetric stretching vibration of C-O, C-C-O, C-O-C bonds, fracture and polymerization [4].

The 3D map of spectra obtained during the thermal curing process can be seen in Figure 3. The datasets imagine obtained spectra during the thermal curing process, with Dataset 0 corresponding to the spectra obtained at 30 °C.

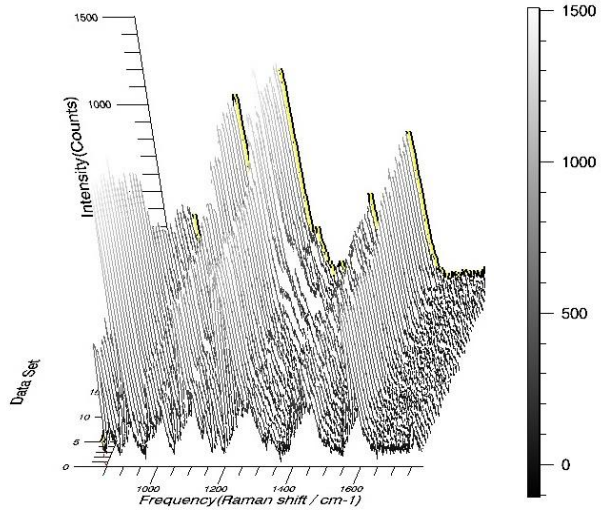

Fig. 3. The 3D map of spectra of the thermal curing process

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Alexandr POŠTA was born in Prague in 1984. In 2017, he received Master of Science degree in engineering at CTU faculty of electronic engineering and started Ph.D. study at Department of Microelectronics.