Microelectronic Engineering 85 (2008) 2276–2281

Contents lists available at ScienceDirect

Microelectronic Engineering

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

Complex permittivity characterization of benzocyclobutene for terahertz applications

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ARTICLE INFO

Article history:
Received 29 January 2008
Received in revised form 19 June 2008
Accepted 6 July 2008
Available online 23 July 2008

Keywords:
Benzocyclobutene polymer (BCB)
Terahertz
Dielectric permittivity
Dielectric loss
Fourier transform spectroscopy
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ABSTRACT

High-quality polymers such as the benzocyclobutene polymer (BCB) provide interesting dielectric feature for terahertz applications. Already used in silicon integrated circuit technologies, this material could become one of the most promising candidates for the realization of future THz waveguides and interconnections on a silicon substrate but also after active devices process on the top of any other technology (GaAs, InP, GaN...). A frequency-dependent complex permittivity of spin-coated thick layers of this low-k dielectric is obtained from transmittance spectra measured with Fourier transform spectroscopy in the frequency range of 0.5–5.4 THz. The dielectric constant and the loss tangent are discussed according to curing conditions of the photosensitive resin used. A low loss tangent value of $7 - 9 \times 10^{-3}$ at 1 THz is obtained with polymerisation in oxygen-free atmosphere. An incomplete curing and a high dose UV exposure have a weak impact on losses. These results associated with the high compatibility of this polymer with silicon and metals make BCB layers well suited for the design of microelectronic THz devices and circuits.

1. Introduction

The terahertz frequency waves offer relatively unexplored applications in microelectronics, the development of innovative imaging and sensing technologies suggests an enormous potential. The instrumentation must be refined to reach these future applications, a primary goal is to characterize usual microwaves media which shows promising feature in the terahertz range. The complex permittivity, including the relative permittivity (ε_r) and the loss tangent ($\tan\delta$) is the basic dielectric property of materials. ε_r and $\tan\delta$ must be well determined in order to build optimal structures guiding or radiating terahertz waves. A method based on the Fourier transform spectroscopy (FTS) is applied to the dielectric characterization of benzocyclobutene polymer (BCB) layers. This polymer is currently one of the more promising dielectric media deposited on semiconductor substrate for terahertz applications.

Polymer dielectric layers and films are already widely used in integrated and microwave circuits because of their good electrical and mechanical properties. Such dielectrics also have been introduced for use in electric micromachines as insulating and interlevel material [1]. Indeed their application field covers an increasingly larger spectrum, such as microelectronics [2] and millimeter-wave applications [3,4]. BCB is an organic polymer which offers good planarization properties, low moisture absorption, and low isotropic dielectric constant and losses [5]. As regards fabrication process, BCB has a good compatibility with various metals. Moreover it is fully compatible with standard semiconductor techniques, and a photosensitive property can be added to increase its processing potential. The deposit procedure consists in spin coating, and the curing requires relatively low temperature (250 °C), which makes it compatible with standard CMOS technologies [2]. Besides, its low permittivity minimizes frequency dispersive phenomena and crosstalk, in particular, by increasing cutoff frequencies of non fundamental modes in waveguides [6]. For planar air/metal layer/dielectric guided structure, the higher the permittivity of the dielectric is, the more the energy is confined in it. In term of antennas, it means that the energy is mostly radiated into the dielectric side. For an elementary dipole antenna, the power ratio between the dielectric and the air can be estimated to about ε_r [7,8]. When this phenomenon becomes problematic, in particular for submillimeter antenna design, the use of lenses becomes interesting [8]. Lens with same material than substrate can be added in order to increase the proportion of the antenna energy which radiates into the lens side, as well as to increase the antenna gain. Alternatively, using a dielectric layer of low permittivity between the substrate and the antenna metallization should better balance the field distribution.

In the THz range, it is still challenging to minimize waveguide attenuation [3] in order to design transmission lines suitable for very low level signals. The low loss properties of the BCB compared

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doi:10.1016/j.mee.2008.07.008

with a low resistivity silicon substrate is exploited here. Indeed, as mentioned above, the insertion of a low-loss low permittivity dielectric layer on silicon substrate based technology allows reducing both the dielectric losses and the substrate coupling [4,9]. Several kinds of planar waveguides can be done on BCB, including microstrip lines, coplanar waveguides and slotlines. The high thickness of a layer deposited allows screening losses of the silicon substrate and does not require high-resistivity silicon (HR-Si) or other substrates. The passive devices of a THz circuit (interconnection, filtering...) could be almost independent from the active part of the circuit, thanks to this layer. Obviously, the idea is to favour the penetration of the electromagnetic field in the BCB rather than in the lossy silicon substrate [9]. This is why, in the microwaves and millimeter wave range, thick layers (>20 μm) contribute to better screen the substrate, and a reduction in the transmission losses is thus observed [4].

The complex permittivity of the dielectric layer should be well determined to design transmission lines and other THz devices. Unfortunately, both the losses in the THz frequency range of most of polymers and their sensitivities to technological process remain badly known [10]. Many factors are involved in losses with the degree of polymerization and of crosslinking, and with the level of moisture, solvent or impurities content. In order to obtain reliable data compatible with THz applications, we need to characterize such layers in their nominal configuration and according to the process conditions used.

Many quasi-optical measurement techniques of the dielectrics parameters over broadband frequency exist [11]. A powerful technique is the Fourier Transform Spectroscopy (FTS) which provides broadband measurements from millimeter wave to ultraviolet wave range, and allows the characterization of a large panel of samples [12]. The relative permittivity ε_r and the loss tangent $\tan\delta$ can be extracted with high precision depending on the thickness and on the permittivity value of the layers. The dispersive FTS (DFTS) could give the two unknown factors (ε_r, $\tan\delta$) in one transmittance phase-shift spectra but in case of low dielectric constant, it needs sufficiently thick layers that are not compatible with few microns to tens of microns desired for THz devices [13]. With the conventional FTS, both the transmission and reflection spectra should be requested to determine the two factors.

In this article, only the transmission spectrum was considered, and measurement is fitted with a frequency-dependent modelling of the layer complex permittivity. The frequency range investigated is between 0.5 and 5.4 THz, and the model is only applied in this frequency range.

The paper is organized as follows. The measurement technique and the samples fabrication are presented in Section II. The permittivity model, the dielectric parameters extraction and the results are exposed in Section III. The complex permittivity is discussed according to samples differing from their process conditions.

2. Measurement method and setup

2.1. Measurement setup

The FTS is performed with an IFS 66v/S Bruker infrared spectrometer (FTIR), based on a standard Michelson interferometer that can reach a resolution of 0.25 cm^{-1}. It can operate in vacuum to reduce the acoustic perturbation and the strong atmosphere absorption lines of the THz range. A first frequency range of 0.5–1.7 THz is covered with a Hg-arc lamp, with a 50 μm and a 100 μm Mylar® beam-splitter and with a liquid-helium cooled silicon-diamond composite bolometer (4.2 K) in rapid-scan mode. Due to the small area of sample to scan, the transmission measurements are magnified with a beam condenser (×5) using ellipsoidal mirrors. The frequency range is extended to 5.4 THz with a thin multilayer Ge/Mylar beam-splitter, with a SiC Globar® source and with a DTGS pyroelectric detector.

2.2. Sample description

The BCB layers deposits onto silicon substrates were carried out in a clean room. The BCB used is based on Cyclotene® 4026-46 available from the Dow Chemical company. The composition of this resin (a B-staged divinylsiloxane-bis-benzocyclobutene oligomer with diazo cross-linkers) allows UV-sensitive properties acting like a negative resin. The thickness layer expected is about 30 μm associated with spin coating speeds of about 600 rpm. These speeds are lower than those specified (1500 rpm) in the processing procedures, which gives 5–15 μm thick layers. Here, the first attempt is to have a larger thickness that facilitates the dielectric parameters extraction rather than a very reproducible thickness and a high planarity. These thicknesses (a few tens of microns) are similar to those of transmission lines realized for very high frequencies [4].

The fabrication process starts with the spin coating of the resin onto a high-resistivity silicon substrate (HR-Si), previously RCA cleaned, and using adhesion promoter. The samples are then soft baked. Due to its light sensitivity the resin is exposed or not to UV light before the final hard cure. Different UV exposure doses have been performed on samples up to 6400 mJ/cm^2 when recommended exposure is about 1800 mJ/cm^2 for a thickness of 30 μm, leading to first crosslinking. In our case no development is done. The wafer is directly placed in oven to perform the final cure. Higher and longer are the temperature and the duration of the cure, and higher is the rate of molecule conversion to benzocyclobutene polymer. According to Dow Chemical specifications [14], the samples should presented a 70% rate with 200 °C during 40 min, and should get nearly complete conversion (95%) with 250 °C during 60 min. We expect lowest dielectric losses with the highest degree of conversion. At the same time, "impurities" must be removed or prevents from chemical linking. The solvent should be completely eliminated with the soft and the hard bake, and the oxidation is avoided with inert N_2 atmosphere rather than air ambience. One sample has been cured in air atmosphere to appreciate this influence.

The 300 μm thick silicon substrate introduces some limitation for the characterisation method. Once the BCB layer cured, 5 × 5 mm^2 squares are etched from the wafer rear side, arranging in membrane some parts the BCB layer, self-supported with the silicon substrate. The silicon etching is performed with an inductively coupled plasma (ICP) technique. Many efforts have been carried out to protect the BCB layer during the etching. Before processing the etching of the substrate, wafers are fragmented into parts, preserving some of them of any physical reaction.

Seven numbered samples are exposed here and main process parameters are gathered in Table 1. The spin coating speed was 600 rpm excepted for sample No. 7 with 800 rpm leading to a thinner BCB layer of 27 μm. With a lower temperature and a lower duration, the hard cure of samples No. 2 and No. 3 are incomplete assuming that molecules conversion is only 85% and 70% respectively. The sample No. 5 is cured in air atmosphere, and samples No. 4, No. 6 and No. 7 have been exposed to UV with a dose of 6400 and 3200 mJ/cm^2. The measurement has been performed on both the BCB layers in membranes and in their silicon substrates.

2.3. Transmission modelling

According to the optical characteristics of measurement setup and the frequency range of interest, the BCB layer is considered as a thin homogenous isotropic (LHI) media into vacuum with flat