Real-time monitoring of light-induced curing of organosilicate glass low-k films by time-domain Brillouin scattering

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Background – Organosilicate glass (OSG) films, developed as low dielectric constant (low-k) insulators for interconnects in advanced ULSI (Ultra Large-Scale Integration) devices, are used to insulate metal conductors and, together with low resistivity metal wires, improve the integrated circuits performance by reducing the signal propagation delay (resistive-capacitive delay) and cross talk noise [1]. Reduction of dielectric constant of OSG films is done by introducing porosities, which are usually obtained by using sacrificial porogens (organic polymers) co-deposited with the silica-like matrix material [1]. The porogens are removed after deposition by thermal annealing, potentially assisted by UV light or electron beam [2]. The control of the curing is of utmost importance for low-k properties and films integration in on-chip interconnects, as porogens curing controls low-k and mechanical properties of the films.

Previously, we showed that the time-domain Brillouin scattering (TDBS) technique is very efficient for nanoscale imaging of porous low-k films [3, 4]. TDBS is a pump-probe technique where the absorption of a fs pump laser pulse launches a coherent acoustic pulse (CAP) that propagates and scatters a time-delayed fs probe laser pulse in a transparent-to-the-probe-wavelength material. The scattered-by-the-CAP light is heterodyned by a reflection of the probe on a stationary surface, giving rise to an oscillating signal. The so-called Brillouin oscillations (BOs) provides information on film properties in the current position of the CAP inside the film, e.g., their frequency is proportional to the product of local optical refractive index (n) and sound velocity (v). In low-k films, TDBS allowed to extract the depth profiles of optical refractive index and longitudinal elastic modulus in partially cured low-k films with remaining porogen residue. Here, we used the TDBS technique for its unique ability to remove porogen residues by the UV probe light action and monitor, at the same (real) time, the changes in Brillouin frequency due to light-induced optical and mechanical property changes associated with the curing.

Methods – Porous organosilicate-based low-k 700 nm-thick films were deposited by a spin-on-glass technique on a Si substrate covered with a 600 nm-thick Al layer. The low-k films were thermally cured at 400°C in air and nitrogen (N) for one or two hours to reach the critical region where films have maximum porosity and lowest dielectric function after the complete porogen removal. This region is crucial for low-k properties because of the matrix re-arrangement leading to the highest quality of low-k films.

The TDBS experimental set-up (Fig. 1(a)) was based on asynchronous optical sampling, where accumulating time delay between green pump and UV probe femtosecond laser pulses is due to difference in the repetition rate of both lasers, allowing fast data acquisition time (5 s/measurement here). The latter allowed real-time monitoring of Brillouin frequency (BF) changes while each sample was exposed to different green pump and UV probe powers.

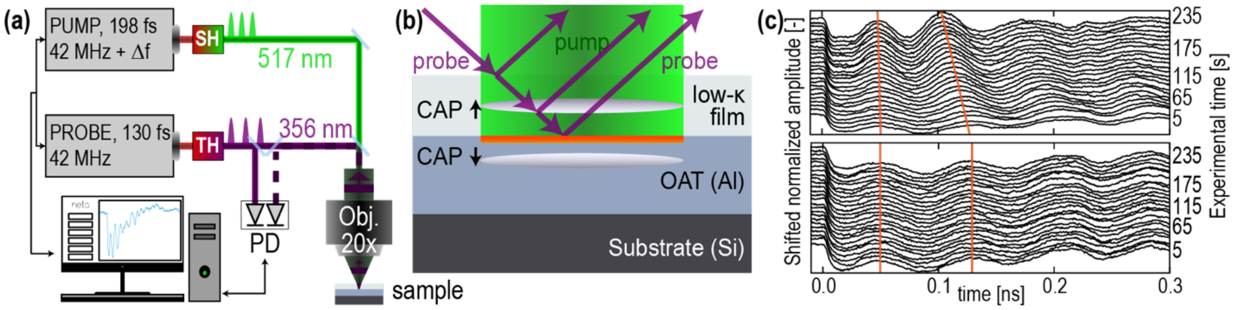

Fig. 1. Schemes of (a) the fast experimental set-up and (b) the TDBS experiment, where the pump absorbed in Al launched CAPs in Al and low-k films where they scatter the probe. (c) BOs period examples vs. the experiment duration in two films annealed for: (top) one hour in N and (bottom) one hour in air.

Results summary and conclusive remarks – As depicted in Fig. 1(c), the BOs period (interval between the two lines) is diminishing with the experiment duration in the film annealed for one hour in N (top), while it is not in that annealed for one hour in air (bottom). The BF increase is a signature of a continuous increase with the experiment duration of the product nv. The TDBS technique allowed to detect extremely small differences existing in the films cured in the critical region where porogen is completely removed but the matrix shrinkage is still not significant. The results also demonstrated local modification of low-k films based on 2-photon absorption, which can be important for atomic layer deposition processes, where the in-situ monitoring ability of TDBS to follow in real time the modification process can be important.

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