Restriction on the laser wavelengths for imaging of metal/epoxy interfaces by time-domain Brillouin scattering

Sandeep S[(1)*], Raetz S[(1)], Nicol E[(2)], Edely M[(2)], Pajusco N[(1)], Chigarev N[(1)], Cuvillier N[(3)], Delozanne J[(3)], Ducousso M[(3)], Tournat V[(1)], Gusev VE[(1)*]

(1) Laboratoire d'Acoustique de l'Université du Mans (LAUM), UMR 6613, Institut d'Acoustique – Graduate School (IA-GS), CNRS, Le Mans Université, Le Mans 72085, France
(2) Institut des Molécules et Matériaux du Mans (IMMM), UMR 6283, CNRS, Le Mans Université, Le Mans, France
(3) SAFRAN TECH, Magny les Hameaux, France

*Corresponding authors' email: sandeep.sathyan@univ-lemans.fr, vitali.goussev@univ-lemans.fr

Background – The time-domain Brillouin scattering (TDBS) is a rapidly developing all-optical experimental technique that uses ultrafast laser pulses to generate and monitor the propagation of coherent acoustic pulses (CAPs) in transparent media. In TDBS, the absorption of pump laser pulses in a metallic layer attached to a transparent sample launches CAPs into the media. The time-delayed probe pulse monitors the CAPs propagation. The imaging and depth profiling capabilities of TDBS have already been demonstrated for various systems, including 3D imaging of biological cells and polycrystalline materials [1].

Here we use TDBS to examine an academically-prepared and an industrial epoxy resin interface with metals such as Al and Ti. Epoxies are the type of reactive polymers used as adhesive or paints in a wide range of industrial applications such as automotive and aerospace. In these applications, the mechanical properties in the vicinity of the metal/epoxy interface play a crucial role in the bond strength and stability of adhesion. The metal/epoxy interface is generally studied using techniques like atomic force microscopy (AFM) and frequency-domain Brillouin scattering (FDBS) microscopy. In AFM, the studies on depth inhomogeneities at nanoscale are destructive/invasive as they require cutting of the sample normally to the surface/interface, whereas FDBS has an axial resolution limited by the probe optical wavelength and has never been applied closer than 10 µm from the metal/epoxy interface [2]. The axial resolution of TDBS imaging is limited by the length of the CAP and can be potentially better than sub-µm scale, i.e., comparable with that of AFM, while it can access the material inhomogeneity below single digit µm distances from the surfaces/interfaces [1,3]. When limited by signal processing the depth resolution of TDBS can be controlled by the acoustic wavelength $\lambda_{ac} = \frac{\lambda_{probe}}{2n}$, where n is the refractive index of the medium and λ_{probe} is the probe laser wavelength. So, by diminishing λ_{probe}, the spatial resolution of TDBS imaging could be enhanced. Yet, our experiments demonstrate that near-UV and visible wavelengths modify the physical properties of the epoxy resin. To our knowledge, the usual FDBS technique used to study the metal epoxy/interface is conducted with visible laser because of its availability in the commercial spectrometers. Our homemade TDBS experimental setup, using near-IR pump and probe lasers, thus enables us to study the metal/epoxy interface non-destructively.

Methods – We implemented TDBS with a fast data acquisition technique based on asynchronous optical sampling (ASOPS). For the generation and detection of the CAPs, we applied femtosecond laser

pulses at pump/probe wavelengths 345/356, 517/535, and 1035/1068 nm. For preparing epoxy academically, we used the combination of diglycidyl ether of bisphenol-A (DGEBA) and diethylenetriamine (DETA). The industrial epoxy used by SAFRAN TECH was the AF 191 structural adhesive film reference of 3M™ Scotch-Weld™. In the DGEBA/DETA case, thin metal film depositions provided opportunity for the CAPs generation in metal and their detection in epoxy from opposite sides (Fig 1a), which helps to reduce pump-related heating of epoxy and where sapphire substrate additionally acts as a heat sink. For AF 191 epoxies prepared on 30 µm-thick aluminium sheets, the pump and probe beams are incident at the metal/epoxy interface from the same side (fig. 1b).

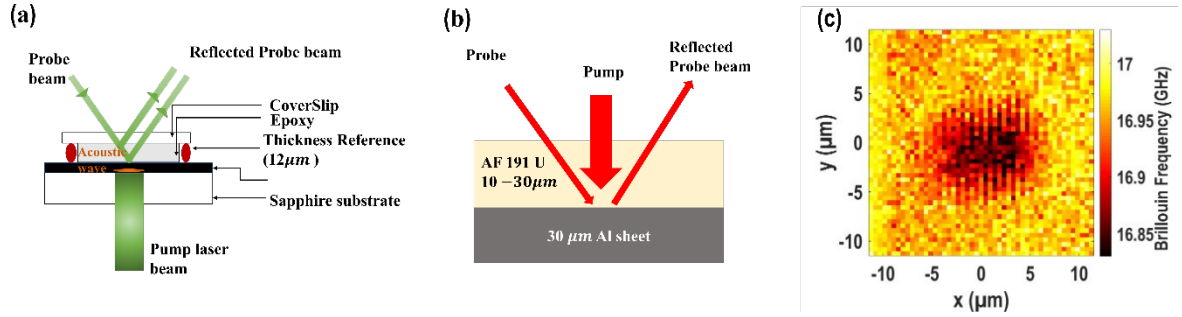

Fig. 1. TDBS experimental configurations in: (a) opposite side, (b) same side. (c) 2D map of residual depth-averaged Brillouin frequency obtained with visible pump and probe wavelength around the position where curing was studied.

Results/Discussion – We observed modification of physical properties of epoxy with the near-UV and visible wavelengths, whereas the experiments with near-IR wavelengths demonstrate the non-destructive probing of the metal/epoxy interface. Fig. 1(c) shows the laser-induced lateral variation of Brillouin frequency around the position where curing is studied with the visible laser. In TDBS experiments, signals are observed up to ~1.5 ns duration, implying GHz CAPs propagation to a depth of ~5 µm from the interface. Analyzing these oscillations as a function of time delay between the pump and probe laser pulses at various lateral locations at the interface produces three-dimensional images of epoxy, which are accumulated during the curing process.

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