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Silicon-on-diamond material by pulsed laser technique

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We present a method to bond directly silicon and diamond plates to obtain a single silicon-on-diamond material, with a carbon–silicon interface of unprecedented quality. The bonding is performed at room temperature, via picosecond 355 nm pulsed laser irradiation of the silicon-diamond interface, through the transparent diamond. The obtained material exhibits excellent mechanical strength and uniformity of the bonding, as shown by mechanical tests and analysis of the cross section based on scanning electron microscopy. The bonding is ascribed to silicon carbide nanolayers at the interface which, along with amorphous silicon nanolayers, have been quantitatively detected and evaluated by means of optical spectroscopy measurements. A physical insight into the processes occurring at the diamond-silicon interface during the pulsed irradiation and cooling has been provided by a finite element numerical model. A rationale is then given for the observed SiC bond in terms of silicon and diamond melting and inter-diffusion. A crucial outcome of the model consists in predicting the effect of the different laser beam parameters on the bonding process, thereby allowing us to obtain a well tailored procedure. An excellent quality silicon-on-diamond is now available for implementing highly integrated electronic devices for diverse application areas, ranging from pixel detectors to biosensors and prostheses for the human body. © *2010 American Institute of Physics.* [doi:10.1063/1.3291043]

In the last ten years the interest in silicon-on-diamond (SOD) material has increased with the relevance of thermal issues in integrated electronics for its close, electrically insulating, contact between diamond and the electronic device semiconductor. Diamond exhibits, as well as other interesting physical properties, from good to excellent thermal properties depending on its state: nanocrystalline/polycrystalline or single crystal. SOD material is generally obtained by the chemical vapor deposition (CVD) growth of diamond onto silicon.[1–4] Studies on direct bonding diamond and semiconductor crystals have also been performed[5–7] with satisfactory results only in the case of GaN-on-diamond.[7] Our technique lies within this latter branch: it uses a simple, noncritical process applicable, unlike the CVD techniques, to any kind of diamond and silicon plates, and, in principle, also to non silicon semiconductors. The growing interest for SOD structures[2,4,8,9] addresses silicon-on-insulator (SOI) devices,[10] but the availability of a versatile technique, independent of material quality, as our method, opens the way to more advanced applications, as monolithic pixel detectors[11–13] and biosensors,[14,15] where signals and elabora-tion electronics can be integrated in rugged, low noise SOD systems

We use UV laser irradiation of diamond and silicon plates under uniaxial compression at room temperature. The radiation transmitted through diamond is absorbed within about ten nanometers of Si, melting locally both materials, which diffuse and solidify in a strong interface bonding. The obtained SODs have been observed by scanning electron microscopy (SEM) and their excellent mechanical adhesion at room temperature has been measured. Micro-Raman and Fourier transform infrared spectroscopy (FTIR) analyses showed SiC and amorphous Si (a-Si) phases at the interface.

We used 500 μm thick polycrystalline CVD diamond (pCVD) (Ref. 16) and $50-300$ μm thick (111) Si plates of 5×5 mm^2 each. The diamond quality is up-to-date but any type of transparent diamond can be used. The samples, carefully cleaned, are inserted into a gas-tight chamber under Ar atmosphere, to avoid oxygen contamination at the interface, and pressed together at ≈ 80 MPa during the irradiation. White light interferometric microscopy has been performed to select the samples smoothest surfaces. The measured roughness heights and widths were, respectively, ~ 10 nm and 100 μm for the best diamond surfaces and about a factor ten lower for the Si ones. Under these conditions we evaluated[17] a surface adhesion greater than 60%, before irra-
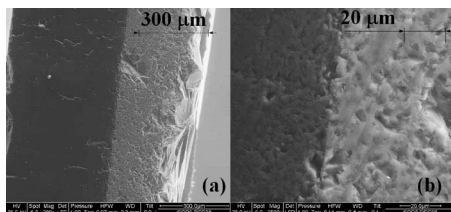
FIG. 1. SEM images of a bonded silicon (right side) diamond (left side) interface. (a) cross section of a sample; (b) enlarged detail.

diation, which was enough to ensure sample bonding.

The 355 nm radiation is provided by a 20 ps Nd:YAG laser with 10 Hz repetition rate. Laser pulses are shot on silicon, through the fused silica viewport of the chamber and the diamond sample. The laser spot on silicon has about 1 mm diameter and $1–10$ J/cm^2 energy density per pulse. The bonding process is performed by manual scanning, with a step chosen such as to illuminate the whole samples interface. Since a single shot was not sufficient to bond the samples, we integrated from 5 to 50 pulses per step. The total process lasted several minutes per sample, but the required time can be decreased by scanning automation.

Optical and SEM analyses of the processed samples showed no evidence of diamond fractures, unlike in results reported for thermally activated bonding methods.[4,5] The sharp and uniform bonding profile is shown, on a micrometer scale, in the SEM micrographs of a sample cross section of Fig. 1.

Tensile resistance tests, performed at room temperature, gave strength lower limits from 5 to 12 MPa due to fracture either of the silicon bulk, away from the interface, or of the external bonds of the SOD faces to the test device. No correlation has been found between mechanical strength and number of pulses.

To investigate the nature of the SOD interface region, Raman measurements were performed, in the range from 100 to 3500 cm^{-1}, by using the Kr 647.1 and 752.5 nm Kr+ laser lines. Presence of a-Si (Ref. 18) was detected in all investigated samples (see Fig. 2). The thickness of the a-Si layer was evaluated by comparing the intensity of the silicon Raman peak at 520 cm^{-1} with that of a broad a-Si band peaked[18] at 480 cm^{-1}. Their ratios are related to the Raman cross-sections and absorption coefficients of the amorphous and crystalline phases.[19,20] The results give thicknesses below 120 nm, with an average value of 85 nm. No dependence
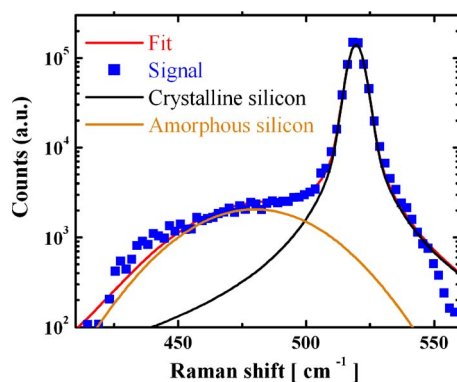

FIG. 2. (Color online) (a) Raman SOD profile (after subtraction of the background) fitted to a sum of a Si pseudo-Voigt profile and an a-Si broad Gaussian, centered at 480 cm^{-1} with a 50 cm^{-1} sigma.
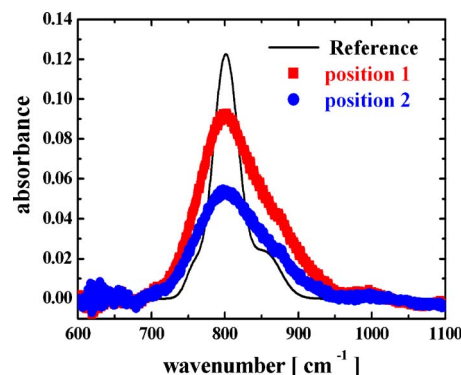

FIG. 3. (Color online) FTIR spectra of two SOD points irradiated with 20 pulses per spot (about 1 J/cm^2 per pulse). The reference spectrum is taken from literature (see Ref. 21).

on the power density (varying over a factor of 2) and the number of pulses (varying over a factor of 10) has been observed.

Transmission properties of SOD samples were investigated by means of FTIR spectroscopy, in the $570–5000$ cm^{-1} range. The obtained SOD sample spectra, have been normalized to the corresponding spectra of an unbonded silicon-diamond couple of equal thickness, in order to obtain the characteristic absorbance of the bonded interface and the depth of the modified interface region. Figure 3 shows two absorption spectra exhibiting a peak at about 800 cm^{-1}, which is assigned to stretching modes of an amorphous or nanocrystalline form of SiC. According to the experimental data provided by FTIR literature,[21] we found that the equivalent thickness of the SiC interface ranges from 50 nm in the samples with the highest number of laser shots (50), down to the sensitivity of the method (3 nm) in case of ten laser shots per position.

SiC and a-Si phases (other than traces of amorphous carbon, detected in some regions of the samples) are signatures of strong heating of the materials in the SOD interface region, during the bonding process. To understand its dependence on the macroscopic parameters (energy density per pulse, laser frequency, and number of laser pulses per position), we developed a numerical model taking into account the physical transformations occurring at the silicon-diamond interface during irradiation. In our experimental conditions, a premelting period with local pressures up to about 1.5 GPa and plasma densities up to 3% of the valence electron concentration, is followed by a silicon melting period, whose temperature increases in few picoseconds up to the free energy equilibrium temperature between diamond and its liquid phase (≈ 4250 K, depending on local pressure).

The details of the model will be expounded in a fore coming paper. We resume here the main results in terms of the energy density thresholds for melting of silicon and carbon as functions of the pulse width, and compare them with the experiments (Fig. 4). The trend of the melting curves is ruled by the prevalence of two different phenomena: the heat diffusion at long pulse widths, the radiation absorption at short widths. The experiments effective in bonding are all above the diamond melting threshold and the only one with an energy density below the silicon threshold has resulted in unbonded Si and diamond plates. Further measurements are needed to validate the curves from the model, however it is clear that the bonding process is based on the solidification
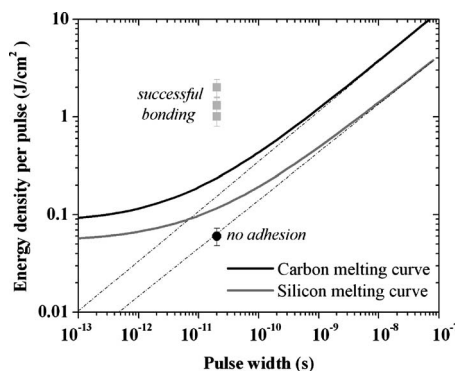
FIG. 4. Calculated energy thresholds for melting of silicon (gray solid line) and carbon (black solid line) as functions of pulse width. Effective bonding experiments are represented by the gray squares, the one yielding no adhesion by the black circle. At large pulse widths the two curves tend asymptotically to a square root trend (diffusion term).

of a fused diamond silicon mixture at the interface and that SiC is its signature. The model does not account for the required number of laser pulses per step since it assumes a flat interface where all points melt, diffuse and solidify all together. In the real world the interface is irregular and it becomes, pulse by pulse, flatter and flatter thereby increasing the surface area which is in contact under the action of the external pressure. This plausible explanation recalls qualitatively the observed dependence of SiC on the number of pulses and indicates the necessity of further investigating the role of the surface roughness on the quality of the SOD interface.

In conclusion, we developed a simple and fast method to bond directly silicon and diamond samples at room temperature. The obtained SOD samples exhibit uniform, defect free profiles on a micrometer scale and a considerable mechanical strength. FTIR analysis evidenced the presence of SiC chemical bond, originated by the melting of sub-micrometer silicon and carbon layers followed by their interdiffusion. Further investigations to optimize the process parameters, which could lead to minimize the interface layer, will be carried on.

The SOD samples of the present work were fabricated using high quality polycrystalline diamond but this choice is not critical as well as the choice of the silicon material. The limited interface modifications and the favorable bonding conditions suggest the use of electronic chip substrates to open ways toward advanced devices such as HEP monolithic pixel detectors and biosensors, (short term), and muscular and neural prostheses (long term).

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