Influence of substrate material on spectral properties and thermal quenching of photoluminescence of silicon vacancy colour centres in diamond thin films

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Nanocrystalline diamond films with bright photoluminescence of silicon-vacancy colour centres have been grown using a microwave plasma enhanced CVD technique. The influence of substrate material (quartz, Al_2O_3, Mo and Si) on a reproducible fabrication of diamond thin films with Si-V optical centres is presented. Film quality and morphology are characterized by Raman spectroscopy and SEM technique. SEM shows well faceted diamond grains with sizes from 170 to 300 nm. The diamond peak is confirmed in Raman spectra for all samples. In the case of the quartz substrate, a redshift of the diamond peak is observed (≈ 3.5 cm^{-1}) due to tension in the diamond film. The steady-state photoluminescence intensity was measured in the temperature range from 11 K to 300 K. All spectra consist of a broad emission band with a maximum near 600 nm and of a sharp zero phonon line in the vicinity of 738 nm corresponding to Si-V centres that is accompanied with a phonon sideband peaking at 757 nm. Activation energies for the thermal quenching of Si-V centre photoluminescence were determined and the effect of the substrate on photoluminescence properties is discussed too.

K e y w o r d s: diamond, Si-V centre, photoluminescence, microwave-plasma enhanced CVD, activation energy

1 Introduction

Colourless diamond is rather rare. Diamonds prepared by chemical vapour deposition (CVD) usually contain nitrogen in different aggregations [1]. A widely studied and applied optical active defect in diamond is the nitrogen vacancy centre (N-V) obtained by nitrogen implantation and annealing [2]. Another optical system is the silicon vacancy centre (Si-V) formed by Si ions incorporated into diamond due to the etching of Si substrates, quartz windows or even bell jars of CVD reactor [3]. Contrary to N-V centres, irradiation and annealing are not needed to promote the formation of Si-V optical centres [4,5]. In diamond CVD films, the localized Si-V centres show spectrally narrow photoluminescence (PL) with a sharp zero phonon line (ZPL) in the vicinity of 738 nm accompanied by a very weak phonon side band. Together with a short lifetime of the excited state about 1 ns [6] and high photostability, Si-V centres make diamond a promising single-photon source [7]. Moreover, since the ZPL position falls into the transparency window of biological tissues in the near infrared spectral region, their luminescence is a suitable tool for bio-imaging [7,8].

For diamond films and nanoparticles, many works were devoted to study the luminescence process activated by Si-V centres [9-11] and the influence of deposition parameters on their PL activity. To achieve PL enhancement and controlled reproducible fabrication, attention was paid among others to the effect of temperature and quality of the substrate material [12-14], composition of deposition gas [13,15], or creation of hybrid structures and arrays [16,17]. Nevertheless, reproducible fabrication of luminescent diamond based material with controllable yield of Si-V centres PL is, despite great progress, still a technological challenge. Valuable information about the luminescence process can be obtained by analysis of the temperature behaviour of Si-V PL in diamond nanoparticles and films [10,11,18].

In this paper, we present a controllable and reproducible method for the fabrication of Si-V centres in nanocrystalline diamond (NCD) films. Moreover, special attention is devoted to finding the correlation between the substrate material and the yield of Si-V colour centres PL. Photoluminescence behaviour is studied in a wide temperature range from 11 to 300 K.

2 Experimental part

Nanocrystalline diamond thin films were grown on quartz, Al_2O_3, molybdenum (Mo), and Si (100) substrates with size up to 10×10 mm^2 and thicknesses 1 mm, 0.6 mm, and 0.5 mm, respectively. Before the CVD growth, substrates were cleaned and ultrasonically pre-treated in a suspension of deionized water and ultra-dispersed detonation diamond powder (diameter 5-10 nm, New Metals and Chemicals Corp. Ltd., Kyobashi). The diamond CVD process was performed in a microwave
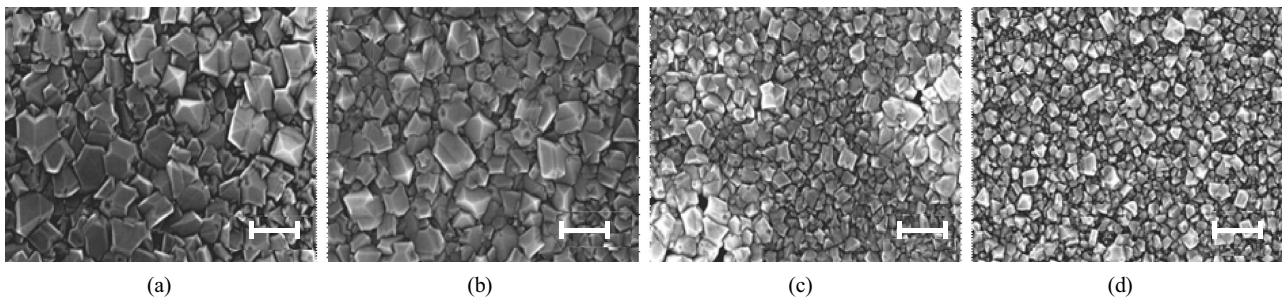
Fig. 1. SEM images of the diamond surface morphology for various types of substrate with scale of 200 nm in all cases: (a) – quartz, (b) – Al_2O_3, (c) – Mo, and (d) – Si
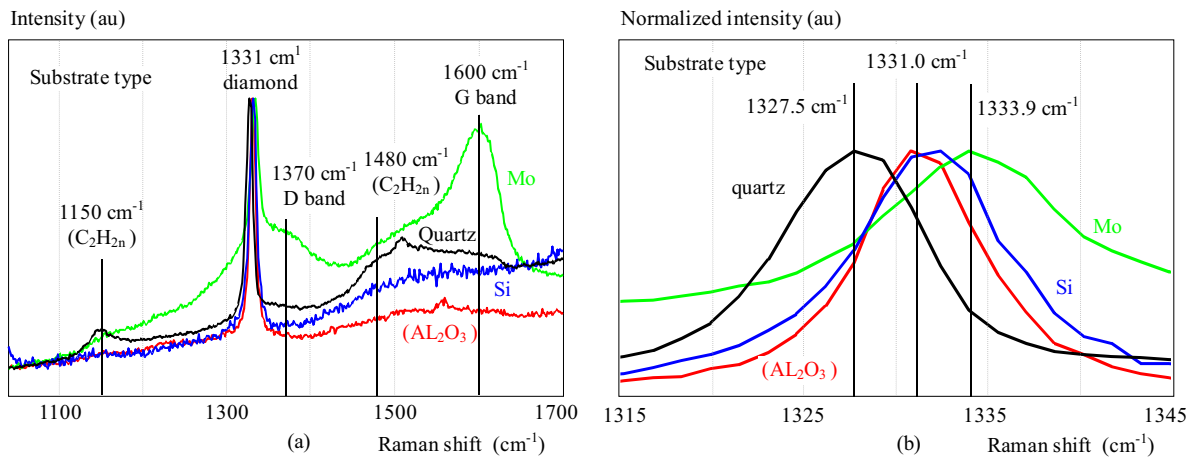

Fig. 2. (a) – Normalized Raman spectra of NCD films grown on various substrate materials, and (b) – detailed view of diamond-peak shift

plasma enhanced CVD reactor using the cavity resonator with focused plasma [19]. All NCD films were deposited together at the same process conditions in hydrogen rich plasma with methane (1% CH_4 with respect to H_2 flow). The substrate temperature TS was $\approx 500\,°C$, total gas pressure 30 mbar, total microwave power 2500 W, and deposition time 8 h.

Surface morphology was characterized by scanning electron microscopy (SEM, e$_L$iNE writer, Raith GmbH). The thickness of diamond films was measured by reflectance interferometry and evaluated by commercial Film Wizard software (Scientific Computing International). The chemical composition and PL activity of Si-V centres in NCD films were studied by Raman and PL spectroscopies at room temperature using a Renishaw In Via Reflex Raman spectrometer with CCD camera and 442 nm excitation wavelength. Temperature dependent emission spectra of photoluminescence excited by a 442 nm line of He-Cd laser were measured in the reflection geometry using a set-up based on a Carl Zeiss SPM2 monochromator [20]. The spectra were taken on the samples fixed to a copper holder of a closed-cycle helium refrigerator (APD Displex SCW-202) within the temperature range 11-300 K. Photoluminescence intensity in the 710-780 nm spectral region was measured with

a cooled RCA31034 photomultiplier (GaAs photocathode) operating in the photon-counting mode.

3 Results and discussions

3.1 SEM

Surface morphology of NCD films deposited on various substrate materials is shown in Fig. 1. For all films, the grains are well faceted with different average sizes, ie 210 nm for quartz, 300 nm for Al_2O_3, 240 nm for Mo, and 170 nm for Si, respectively. Evaluated film thicknesses 230 nm for quartz, 150 nm for Al_2O_3, 140 nm for Mo, and 70 nm for a silicon substrate are different as well. This difference is related mainly to four factors: (i) various delay times caused mainly by substrate diffusion coefficient against carbon atoms and formation of a carbide film, (ii) different nucleation efficiencies (*ie* substrate surface energies, nucleation densities, *etc*), (iii) different early stage diamond growth phases (*ie* formation of interlayer, etching of substrate, surface migration, *etc*), and (iv) different substrate thicknesses (*ie* different substrate temperature given by distance to plasma) [21,22].
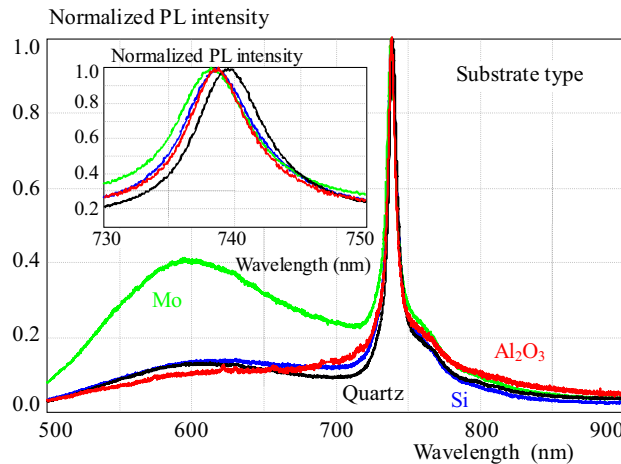
Fig. 3. Normalized room-temperature PL emission spectra of NCD films grown on various substrate materials with PL excitation wavelength 442 nm

3.2 Raman spectroscopy

Figure 2 shows Raman spectra of NCD thin films grown on various substrate materials. All Raman spectra consist of a pronounced diamond peak centred near ≈ 1331 cm^{-1}. For quartz and Mo substrates additional four peaks centred at 1150 cm^{-1}, 1370 cm^{-1}, 1480 cm^{-1} and 1600 cm^{-1} are well recognized.

The bands at 1150 cm^{-1} and 1480 cm^{-1} are attributed to trans-polyacetylene groups (C-H bonds) [23]. An inconspicuous sub-band at the vicinity of 1150 cm^{-1} typically occurs in nano- or ultrananocrystalline diamond films. Bands at 1370 cm^{-1} and 1600 cm^{-1} represent D and G bands [24] corresponding to sp^2 hybridised carbon phase. The bands at 1480 cm^{-1} and 1600 cm^{-1} converge to a broad band extended from 1430 cm^{-1} to 1640 cm^{-1} with the most pronounced structure for Mo and quartz substrates. In agreement with SEM images, the sp^2 hybridized phase is ascribed to the content of grain boundaries and graphitization [13]. Moreover, a shift of diamond peak position is observed in Fig. 2b. The largest shift was observed for the quartz substrate (about 3.5 cm^{-1}). This effect is also manifested by luminescence spectra shown in Fig. 3 and it is attributed to the stress induced at the interface. The stress appears in CVD process when the samples are cooled down from the deposition temperature to the room temperature. The magnitude of induced stress is affected by deposition conditions and diamond film properties (morphology, thickness) but depends mainly on the mismatch of the thermal and mechanical properties between substrate and diamond film [25,26].

3.3 Photoluminescence

Normalized PL emission spectra of NCD films simultaneously grown on various substrate materials are shown in Fig. 3. All spectra consist of a broad emission band with a maximum near 600 nm and of a sharp zero phonon line (ZPL) near 738 nm accompanied with phonon sideband peaking at 757 nm. The ZPL originates from electronic

transition $^2E_u \rightarrow {^2E_g}$ in Si-V centres [11] and for all substrates, ZPL spectral width is about 6 nm. In the inset of Fig. 3, we can see subtle ZPL shift, which is the most pronounced for the quartz substrate. As mentioned above, this effect could be assigned to built-in stress in the NCD film caused by the mismatch of thermal expansion coefficients.

The broadband luminescence peaking at ≈ 600 nm is attributed to structural defects connected with the presence of amorphous sp^2 hybridized carbon [27,28]. For the Mo substrate, the broadband luminescence is well pronounced and the most intensive relative to ZPL one (2.5 for intensity ratio and 0.15 for integral intensity ratio). This finding correlates well with the Raman spectrum which exhibits the highest intensity at 1600 cm^{-1}. For all other substrates, the ratio of the broad band emission intensity to the ZPL intensity reveal almost the same value (7.5 for intensity ratio and 0.4 for integral intensity ratio). It must be emphasized that no silicon-containing gas mixtures were used during depositions, so Si atoms were incorporated into the NCD films as a result of chemical etching of exposed Si-containing substrates and components of CVD reactor chamber (quartz bell jar) [10].

Figure 4 shows the ZPL integral intensity and the ratio of diamond/non-diamond carbon phase areas for various substrate materials. Although diamond/non-diamond phase ratios are almost comparable in NCD films deposited on Al$_2$O$_3$ and Si substrates, film grown on the Al$_2$O$_3$ substrate demonstrates poor ZPL intensity, whereas the ZPL integral intensity in the film grown on a Si substrate is several times higher. For NCD grown on a quartz substrate, the ratio of diamond/non-diamond is lower, but PL integral intensity reaches the maximum. With a further drop of the ratio, for the NCD film on molybdenum substrate, integral ZPL intensity decreases again.

Figure 5 shows the temperature dependence of Si-V photoluminescence integral intensity over 11-300 K temperature range. The observed intensity development was described by the Boltzmann thermally activated process
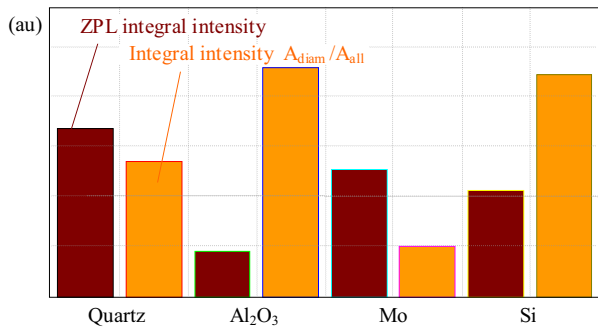
Fig. 4. ZPL integral intensity (full bars) and the ratio of areas of Raman peaks corresponding to diamond and non-diamond phase (empty bars) in NCD films deposited on the various substrate material
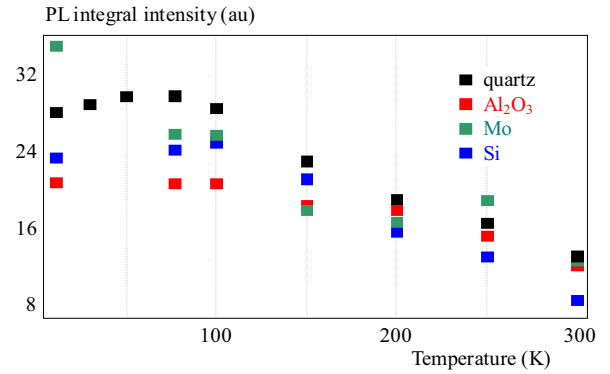

Fig. 5. Temperature dependence of integral intensity for ZPL of Si-V centre photoluminescence for NCD film grown on quartz, Al_2O_3, Mo, and Si substrate, with the latter three integral intensities multiplied by factor of 2
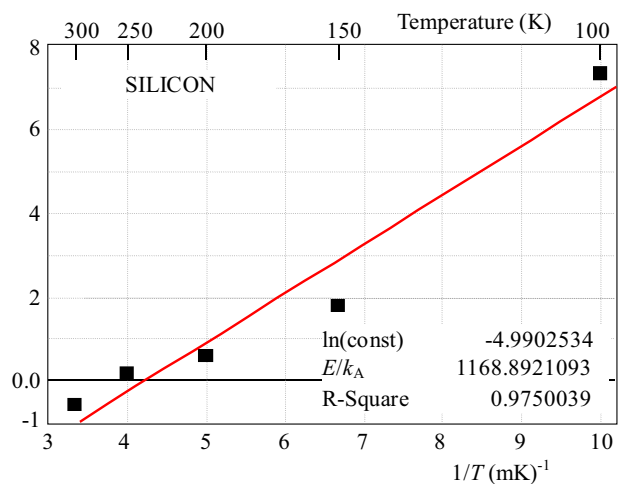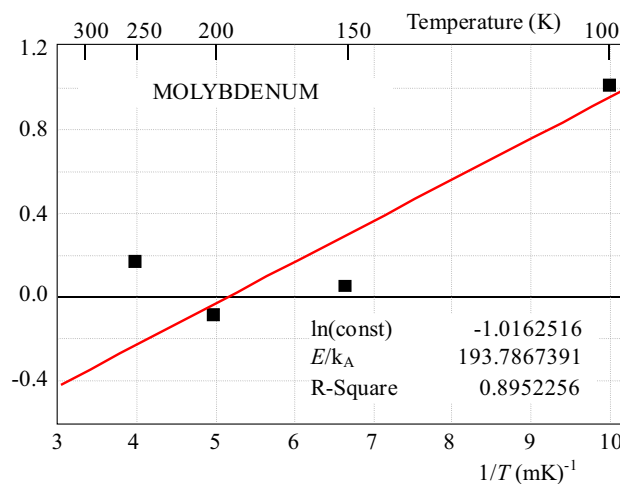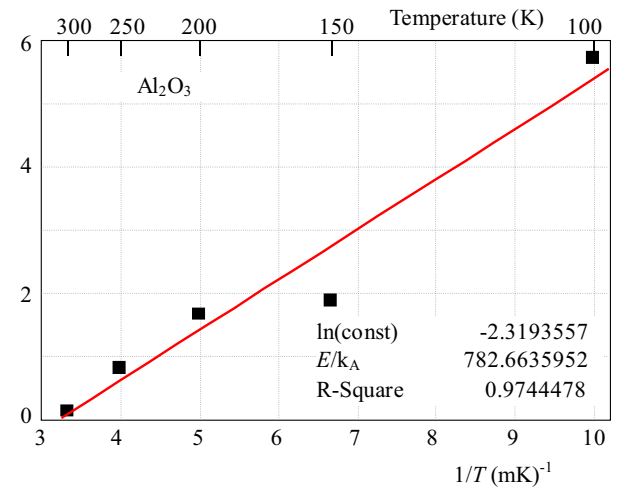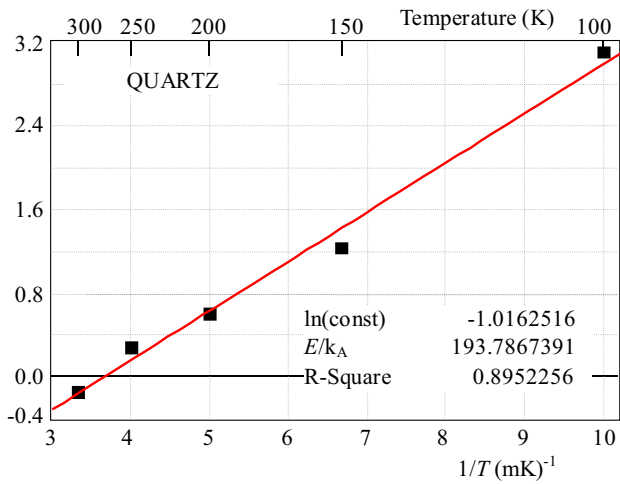

Fig. 6. Linear fit of logarithmic plot of $I(0)/I(t)$ *vs* $1/T$ for NCD films deposited on quartz, Mo, Si, and Al_2O_3 substrates (squares)

[28] with thermal activation energy needed for recombination of photo-carriers from the excited state by non-radiative processes. During analysis, the broadband emission was subtracted from the spectra to eliminate the influence of broadband emission on the ZPL intensity temperature dependence. Integral intensity of ZPL slightly increases up to the maximum at the temperature of 100 K

for quartz, Al_2O_3 and Si substrate in agreement with the study of Feng *et al*, [18]. For molybdenum substrate, the ZPL integral intensity decrease is observed. Next, the ZPL intensity behaviour follows a steeper decrease in the region from 100 K to room temperature and exhibits similar nature for all studied samples.

The value of activation energy of thermal quenching, *ie* energy needed for occurrence of concurrent non-radiative processes, was calculated by the method proposed in [18,28,29]. Figure 6 shows the behaviour of function $\ln(I(0)/I(T))$ *vs* $1/T$ calculated for all studied samples and temperatures higher than 100 K. Quantity $I(0)$ stands for a theoretical value of PL intensity at 0 K. The slope of lines determine the value of PL thermal quenching activation energy E_A. Its determination is problematic and generally not discussed in detail in available literature. Thus, we opted for the same approach as in the work of Collins *et al* [29] and for all cases we selected the maximal intensity in temperature dependence as the value of $I(0)$. By this method the approximate values of activation energy were determined as follows: 40 meV for quartz substrate, 70 meV for Al_2O_3 substrate, 20 meV for Mo substrate, and 100 meV for Si substrate. Values obtained for The NCD films grown on silicon and Al_2O_3 substrates corresponded with results reported in refs [18,28,30].

3.4 Discussion

Data in Fig. 3 and 4 might indicate that the photoluminescence activity of Si-V centres, ie creation of Si vacancy pairs, is influenced by the quality of the diamond film. On one hand, these results are influenced by grain sizes. Incorporation of vacancies into NCD is due to non-equilibrium of the growth process and the presence of other defects, *eg* grain boundaries and sp^2 phase [31]. On the other hand, only a few percent of vacancies are formed on the surface of CVD diamond [32].

The influence of different sp^2 carbon phase ratio in our films on PL spectra development could be reflected in two ways. First, for intensive Si-V photoluminescence, a certain ratio of non-diamond carbon phase is needed, as was shown in [13], it relates with the creation of silicon-vacancy [1]. Second, sp^2 phase introduces continuum of states into the gap. Because Si-V centre luminescence is not excited resonantly in our case, these continua might act as absorption centres collecting excitation energy, which is consecutively transferred to the Si-V centre. Thus, with increase of sp^2 phase content, rise in PL intensity should be expected due to increasing amount of excitation energy absorbed and transferred to Si-V centre. These effects correspond with results presented in Fig. 4. The film on Al_2O_3 substrate has the highest diamond quality, thus the lowest density of absorbing centres and probably the lowest concentration of Si-V pairs. For film on Si substrate, similar quality was reached, but due to the Si-containing substrate one should assume higher concentration of Si-V centres. Maximal Si-V ZPL intensity for film on quartz should be result of the optimal amount of non-diamond carbon phase and concentration of Si-V centres, again supported by Si-containing substrate. In similar line of arguments, we assume that ZPL intensity obtained for film on Mo substrate is rather result of the highest amount of sp^2 phase.

The extraordinary low-temperature part of the ZPL integral intensity dependence (<100 K) in Figure 5, observed for NCD films deposited on quartz and Mo substrates, could be explained by the pronounced effect of other optically active centres in diamond film. Since, in addition to the contribution of Si-V centres, all photoluminescence emission spectra in Fig. 3 also consist of a broad band with a maximum near 600 nm, at least two mutually competitive PL processes take place at an excitation wavelength of 442 nm. The expected temperature dependence of the ratio of absorbed excitation energy transferred to these centres could lead to the observed behaviour of ZPL integral intensity at temperatures below 100 K. Besides the room-temperature PL spectra in Figure 3, where the broad band is the most evident for films on quartz and Mo substrates, this explanation is also supported by corresponding Raman spectra in Fig. 2. Contrary to works [18,28], where a nearly constant value of ZPL integral intensity up to 100 K was seen, the ZPL integral intensity shows a different trend at low temperatures for films on substrates where pronounced D and G bands were observed.

The different values in activation energies of thermal quenching could be explained again by the presence of different amounts of non-diamond phases, as well as by differences in NCD films lattice constants on various substrates. The higher amount of non-diamond phases decreases the distance between the absorbing centres which can result in a higher probability of non-radiative energy transfer between them and in a decrease of the activation energy of PL thermal quenching. In addition, the activation energy can be reduced by modification of the structure of energy levels due to stress originated from differences between the lattice constants of diamond and substrate. Due to a very similar stress reflected in the position of the diamond peak in Raman spectra, Fig. 2(b), we conclude that the amount of sp^2 phase predominantly affects the luminescence process for NCD films on Al_2O_3 and Si substrates. Since the ratios of non-diamond carbon phase is comparable, as we can see in Fig. 4, a similar energy transfer could be expected, thus similar activation energies of thermal quenching. The different calculated values could partly result from an inaccuracy of applied $I(0)$ connected with its difficult determination [29] from the extraordinary behaviour of the low-temperature part of the temperature dependence. For NCD films on Mo and quartz, stress should be a dominant factor. From Fig. 4, it could be concluded that non-diamond content contribution into luminescence process is stronger for the film on Mo substrate than on a quartz substrate. So we expect a higher activation energy for film on quartz substrate.

4 Conclusions

Nanocrystalline diamond thin films grown on various substrates were analyzed using steady-state photoluminescence, Raman scattering, and SEM measurements. Substrate material influenced the seeding and CVD film

growth, and concentration of PL active Si-V centres within the film. The grown NCD films exhibited built-in stress, most pronounced for quartz substrate, as indicated by the shift of the diamond Raman peak and by PL measurements. PL of all NCD samples was dominated by the zero phonon line of Si-V centre emission. Thus, PL activity of Si-V centres independent of various substrate material was demonstrated. The PL of all studied samples was temperature sensitive with a similar development of ZPL integral intensity from 100 K to 300 K. This temperature development is consistent with a Boltzmann thermally activated process with activation energies for the nonradiative recombination processes of 40 meV for the quartz substrate, 70 meV for the Al_2O_3 substrate, 20 meV for the Mo substrate, and 100 meV for the Si substrate. The luminescence properties originate partly from the electronic transition in the Si-V centre and partly from transfer of excitation energy absorbed by structural defects due to the presence of sp^2 hybridized carbon phase.

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