



# Isotopic fingerprints of Pt-containing luminescence centers in highly enriched $^{28}\text{Si}$

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Recently we have shown that the reduction in the photoluminescence linewidth of many deep luminescence centers in highly enriched  $^{28}\text{Si}$  results in well-resolved isotopic fingerprints. This allows for a better characterization of a defect center, as not only the involvement of a specific element but also the number of atoms of that element within the complex can be determined. Surprisingly, we have found that many well-known luminescence centers have a different composition than originally supposed. In addition, we have found a large number of four- and five-atom luminescence centers involving the elements Cu, Au, and Li. Here we introduce series of four- and five-atom deep luminescence centers involving a single Pt atom together with Cu and Li, similar to what has been seen previously for Au-containing luminescence centers.

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## I. INTRODUCTION

Recently, optical studies of isotopically enriched  $^{28}\text{Si}$  have given rise to a number of unforeseen results due to the significantly reduced spectroscopic linewidths which result from the elimination of inhomogeneous isotope broadening. These results span the fields of shallow<sup>1-3</sup> and deep<sup>4</sup> impurity infrared-absorption spectroscopy, shallow bound exciton (BE) photoluminescence (PL) and photoluminescence excitation spectroscopy,<sup>2,5,6</sup> and deep center PL spectroscopy.<sup>7-10</sup> For the deep PL centers, often referred to as isoelectronic BE due to their relatively large PL efficiency compared to shallow donor and acceptor BE in Si, these reduced linewidths have resulted in a new and powerful method of characterizing the constituents of the binding center.<sup>7-10</sup> While isotope shifts have been an established method for the investigation and identification of such deep PL centers for many years,<sup>11</sup> the observed shifts of the PL lines have typically been smaller than the spectroscopic linewidths, revealing the participation of a given element in the center but not the number of atoms of that element. By making use of the dramatically narrower spectroscopic linewidth of no-phonon (NP) PL transitions in isotopically enriched  $^{28}\text{Si}$  together with a high-resolution spectrometer, PL peaks due to combinations of different isotopic masses of the constituents of a complex can be fully resolved, resulting in an "isotopic fingerprint" of the defect constituents.

Here we apply this method to investigate Pt-related deep luminescence centers in Si. Two Pt centers, in particular, have been discussed previously: the so-called 777 and 884 meV Pt centers. Originally they were thought to be Fe related<sup>12</sup> but later studies involving the  $^{195}\text{Au}/^{195}\text{Pt}$  radioiso-

tope decay pair found evidence of Pt playing a major role in these centers<sup>13-15</sup> and they were hence labeled Pt-Fe centers. The fact that the 778 meV  $\text{Ag}_4$  center<sup>8</sup> is very close to the 777 meV Pt-related center has led to some confusion<sup>13</sup> but we can confirm the existence of both centers. While we previously have shown the involvement of four and three Cu atoms in the 777 and 884 meV centers, respectively,<sup>10</sup> we can now confirm that only a single Pt atom is contained in these centers, which we therefore label  $\text{Cu}_4\text{Pt}$  and  $\text{Cu}_3\text{Pt}$ , respectively. In addition to these two centers we introduce a series of four-atom  $(\text{Cu},\text{Li},\text{Pt})_4$  centers and a series of five-atom  $(\text{Cu},\text{Pt},\text{Li})_5$  centers, similar to the four- and five-atom centers reported earlier for Au.<sup>10</sup> We did not observe the often mentioned 1026 meV Pt-related center<sup>15</sup> in our experiments.

## II. EXPERIMENTAL METHOD

All of the results presented here use the same highly enriched (99.991%)  $^{28}\text{Si}$  material which has produced the narrowest linewidths in our previous studies.<sup>6,8</sup> To prepare samples containing only  $^{195}\text{Pt}$ , discs of  $^{28}\text{Si}$  were implanted with  $^{195}\text{Hg}$  at 60 keV with a dose of approximately  $10^{13} \text{ cm}^{-2}$  using the ISOLDE facility at CERN.  $^{195}\text{Hg}$  decays rapidly to  $^{195}\text{Au}$ , which in turn decays to  $^{195}\text{Pt}$  with a half life of 186 days. While the Hg conversion was complete before the PL experiments were begun, these samples contained both Au and Pt isotopes. Another sample was prepared by implanting  $^{194}\text{Pt}$ ,  $^{195}\text{Pt}$ , and  $^{198}\text{Pt}$  at 60 keV with a dose of approximately  $10^{14} \text{ cm}^{-2}$ . The implanted samples were annealed at 950 °C for 10–30 min under flowing Ar to both

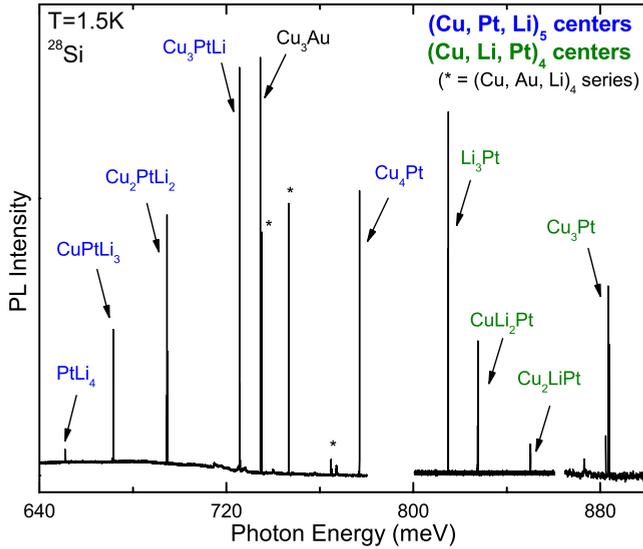


FIG. 1. (Color online) Overview of the four-atom (Cu, Au, Li)<sub>4</sub> and the five-atom (Cu, Li, Au)<sub>5</sub> series in <sup>28</sup>Si at low resolution. Peaks labeled with a \* are Au related centers (Ref. 10) (the <sup>195</sup>Pt resulted from the decay of <sup>195</sup>Au, some of which remains in the sample).

remove the implantation damage and to allow the Au or Pt to diffuse into the bulk of the sample. A third sample was prepared by diffusion of natural Pt for 24 h at 1000 °C in a sealed quartz ampule, evacuated, and backfilled with Ar. Cu and Li were then introduced by heating the sample to ~700 °C in a quartz tube together with the desired impurity. After all annealing or diffusion processes the sealed ampules or samples were quenched to room temperature in methanol, and the samples were then cleaned and etched in HF/HNO<sub>3</sub>. In some cases the samples were later requenched directly into methanol after a quick heating to ~700 °C. All samples and quartzware were cleaned in a KCN solution to remove metallic surface contaminants prior to any diffusion or annealing treatments.

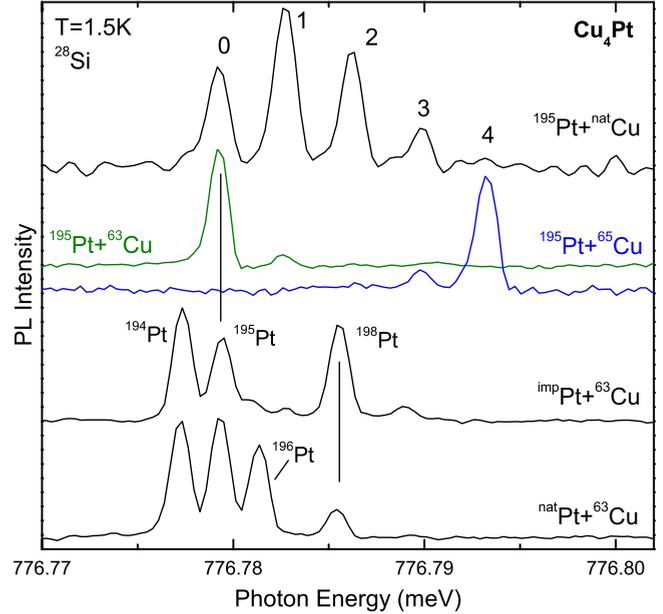


FIG. 2. (Color online) The 777 meV Cu<sub>4</sub>Pt system. The upper spectrum shows a sample diffused with <sup>nat</sup>Cu. Labels 0–4 denote the number of <sup>65</sup>Cu in the complex. Below this, two spectra show samples with monoisotopic Cu. The bottom two spectra show samples that have been either implanted with Pt isotopes or diffused with natural Pt. The relative intensities of the PL lines in the <sup>nat</sup>Pt spectrum closely resemble the isotope abundances of natural Pt. All spectra recorded at 1.5 K and resolutions better than 1.4 μeV.

The impurities diffused into the samples were <sup>nat</sup>Cu (69.2% <sup>63</sup>Cu+30.8% <sup>65</sup>Cu), <sup>nat</sup>Li (7.6% <sup>6</sup>Li+92.4% <sup>7</sup>Li), and enriched single isotopes: 99.8% <sup>63</sup>Cu, 99.6% <sup>65</sup>Cu, 95.5% <sup>6</sup>Li, and 99.99% <sup>7</sup>Li. The natural abundances of <sup>192</sup>Pt, <sup>194</sup>Pt, <sup>195</sup>Pt, <sup>196</sup>Pt, and <sup>198</sup>Pt are 0.8%, 33.0%, 33.8%, 25.2%, and 7.2%, respectively. All diffusants were high-purity materials. It should be noted that some Cu was always detected due to the difficulty in eliminating trace Cu contamination. While this unintentional Cu accurately reflected the <sup>nat</sup>Cu

TABLE I. Energies of all identified Pt-related complexes including all significant electronic states at 1.5 K and high resolution. All energies *E* are in meV for peaks of <sup>6</sup>Li, <sup>63</sup>Cu, and <sup>195</sup>Pt. The relative intensities for the different electronic states are extracted from low resolution files at 4.2 K. Errors are smaller than 0.01 meV. Label † from Ref. 13.

Complex	<i>E</i> (meV)	Rel. intensity	Complex	<i>E</i> (meV)	Rel. intensity
PtLi <sub>4</sub>	650.918	1	Li <sub>3</sub> Pt	814.782	0.03
PtLi <sub>4</sub>	651.056	0.05	Li <sub>3</sub> Pt	814.882	1
CuPtLi <sub>3</sub>	671.291	0.01	CuLi <sub>2</sub> Pt	827.451	0.06
CuPtLi <sub>3</sub>	671.643	1	CuLi <sub>2</sub> Pt	827.569	1
CuPtLi <sub>3</sub>	671.647	0.03	Cu <sub>2</sub> LiPt	849.962	0.08
Cu <sub>2</sub> PtLi <sub>2</sub>	694.571	0.20	Cu <sub>2</sub> LiPt	850.131	1
Cu <sub>2</sub> PtLi <sub>2</sub>	694.613	1	Cu <sub>2</sub> LiPt	851.192	0.18
Cu <sub>3</sub> PtLi	725.599	1	Cu <sub>3</sub> Pt	882.360	0.22
Cu <sub>3</sub> PtLi	725.829	0.13	Cu <sub>3</sub> Pt	883.432	1
Cu <sub>4</sub> Pt	776.779	0.01	Cu <sub>3</sub> Pt	883.792	0.86
Cu <sub>4</sub> Pt {A}†	776.926	1			

TABLE II. The observed Li, Cu, and Pt isotope shifts of the no-phonon lines of the centers studied here, in  $\mu\text{eV}$  per amu.

Complex	$E$ (meV)	Li	Cu	Pt
PtLi <sub>4</sub>	650.918	41		3.1
CuPtLi <sub>3</sub>	671.643	46	4.1	3.6
Cu <sub>2</sub> PtLi <sub>2</sub>	694.571	51	4.3	2.8
Cu <sub>3</sub> PtLi	725.599	86	2.9	2.8
Cu <sub>4</sub> Pt	776.779		1.7	2.1
Li <sub>3</sub> Pt	814.882	52		4.2
CuLi <sub>2</sub> Pt	827.569	25	5.8	3.0
Cu <sub>2</sub> LiPt	850.131	70	2.3	2.9
Cu <sub>3</sub> Pt	882.360		3.6	2.2

abundances before enriched Cu diffusions were performed, it later showed slightly different ratios due to contamination of the furnace and quartzware. When diffusing a single isotope into a sample we have in most cases reached a very high degree of enrichment, especially for Cu and <sup>7</sup>Li, where the starting materials were very highly enriched, and in the case of <sup>7</sup>Li, any contamination with background <sup>nat</sup>Li is already mostly <sup>7</sup>Li. However, a fine balance between diffusing a sufficient amount of one isotope and oversaturating the sample had to be found. In the case of <sup>6</sup>Li, a considerable amount of <sup>7</sup>Li remained visible in most spectra due to both the lower enrichment of the starting material and the dominance of <sup>7</sup>Li in any contamination from background <sup>nat</sup>Li. Hence the <sup>6</sup>Li enrichment in our samples sometimes only amounts to ~80%.

A Bomem DA8 Fourier transform spectrometer was used to obtain the PL spectra with instrumental resolutions between 2.5 and 0.6  $\mu\text{eV}$ . The samples were loosely held in superfluid helium at a temperature of 1.5 K. The PL spectra were collected using bulk excitation at 1047 nm and a liquid nitrogen cooled Ge photodetector. The NP transition energies in <sup>28</sup>Si differ slightly (downshifted by about 62  $\mu\text{eV}$ ) from those in <sup>nat</sup>Si, which can be related to the difference in band-gap energy.<sup>2</sup>

### III. RESULTS AND DISCUSSION

Two of the deep luminescence centers discussed here have been shown before to contain Pt, namely, the centers referred to as the 777 and 884 meV centers.<sup>13–15</sup> We will first discuss these and then focus on the newly discovered Pt complexes. We have reported on the 777 and 884 meV centers recently<sup>10</sup> but the number of Pt atoms involved was unclear at that time.

Figure 1 shows all discussed deep centers at low resolution. We can see the newly discovered four-atom (Cu, Li, Pt)<sub>4</sub> and the five-atom (Cu, Pt, Li)<sub>5</sub> series. It should be pointed out that the Pt series introduced here seem very similar to the Au series introduced earlier.<sup>10</sup> However, there are major differences: while for Au centers the five-atom centers have a smaller exciton binding energy (higher PL energy) than the four-atom centers, for Pt the five-atom cen-

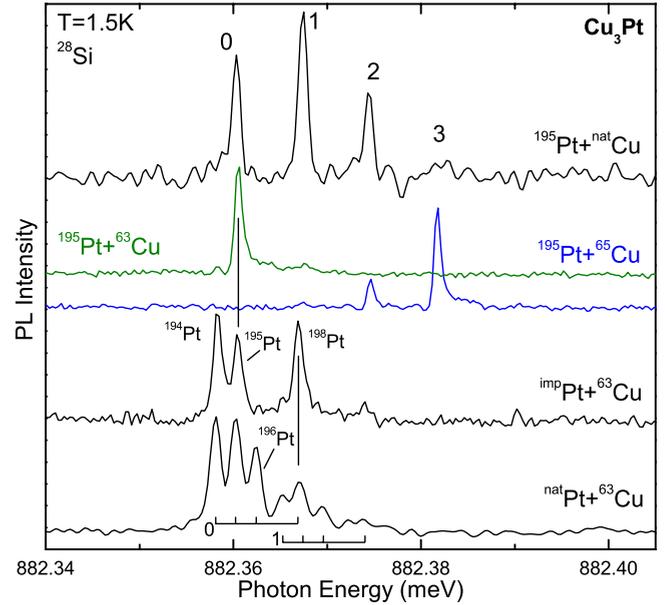


FIG. 3. (Color online) The 884 meV Cu<sub>3</sub>Pt system. The upper spectrum shows a sample diffused with <sup>nat</sup>Cu. Labels 0–3 denote the number of <sup>65</sup>Cu in the complex. Below this, two spectra show samples with monoisotopic Cu. The bottom two spectra show samples that have been either implanted with Pt isotopes or diffused with natural Pt. The <sup>nat</sup>Pt, <sup>63</sup>Cu sample also contains a small amount of <sup>65</sup>Cu, slightly obscuring the spectrum. All spectra recorded at 1.5 K and resolutions better than 1.4  $\mu\text{eV}$ .

ters have a larger binding energy (lower PL energy) than the four-atom centers. Similarly the exciton binding energy decreases for Au centers with increased Li content while it increases for the Pt centers.

#### A. Cu<sub>4</sub>Pt complex

We have now obtained samples containing several different Pt isotopes and can thus determine the number of Pt atoms in all centers studied here to be one, similar to the Au centers introduced earlier.<sup>10</sup> Figure 2 shows the 777 meV center in <sup>28</sup>Si for three samples with different Cu isotope content, demonstrating the involvement of four Cu atoms. We use the same labeling scheme for all Cu-containing centers studied here: “0” refers to no <sup>65</sup>Cu, “1” refers to one <sup>65</sup>Cu atom, etc. In Fig. 2 the lines are labeled with numbers from 0 to 4, so the lowest energy line is due to complexes with four <sup>63</sup>Cu atoms. By replacing the lighter isotopes with <sup>65</sup>Cu one by one the highest energy line is reached, which is due to four <sup>65</sup>Cu atoms. In the upper spectrum shown in Fig. 2 the intensities of these five lines reflect the natural abundance of the two stable copper isotopes in a four-atom cluster. The two spectra with monoisotopic Cu clarify the labeling scheme. The second spectrum from the bottom shows a sample containing only <sup>63</sup>Cu isotopes together with the Pt isotopes <sup>194</sup>Pt, <sup>195</sup>Pt, and <sup>198</sup>Pt while the bottom most spectrum is obtained from a sample diffused with <sup>nat</sup>Pt and <sup>63</sup>Cu. This results in a pattern of PL lines that reflect the relative intensities of the different isotopes. While the implanted sample displays a pattern characteristic to this implantation,

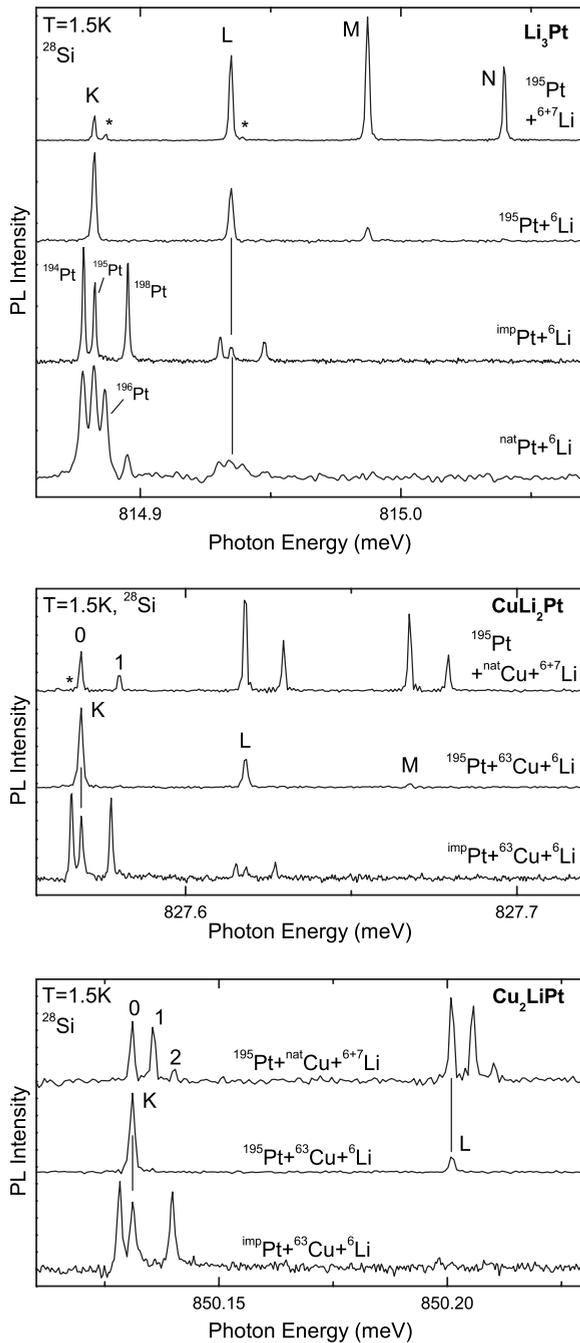


FIG. 4. Spectra of the four-atom  $\text{Cu}_x\text{Li}_{(3-x)}\text{Pt}$  series in  $^{28}\text{Si}$ . Numbers from 0 to 3 designate the number of  $^{65}\text{Cu}$  atoms in the cluster, letters K, L, M, and N designate the number of  $^7\text{Li}$  atoms in the cluster. Peaks labeled with a  $\star$  are of a different, overlapping electronic state. All spectra recorded at 1.5 K and resolutions better than  $1.4 \mu\text{eV}$ .

which we will see repeated for all other PL centers discussed here, the diffused sample at the bottom shows the natural abundances of Pt isotopes with an energy spacing that scales according to isotope mass differences. The measured relative intensities are 34%, 34%, 25%, and 7% for  $^{194}\text{Pt}$ ,  $^{195}\text{Pt}$ ,  $^{196}\text{Pt}$ , and  $^{198}\text{Pt}$ , respectively, closely following the natural abundances. This makes clear that only a single Pt atom is involved in the formation of this center since if there were two

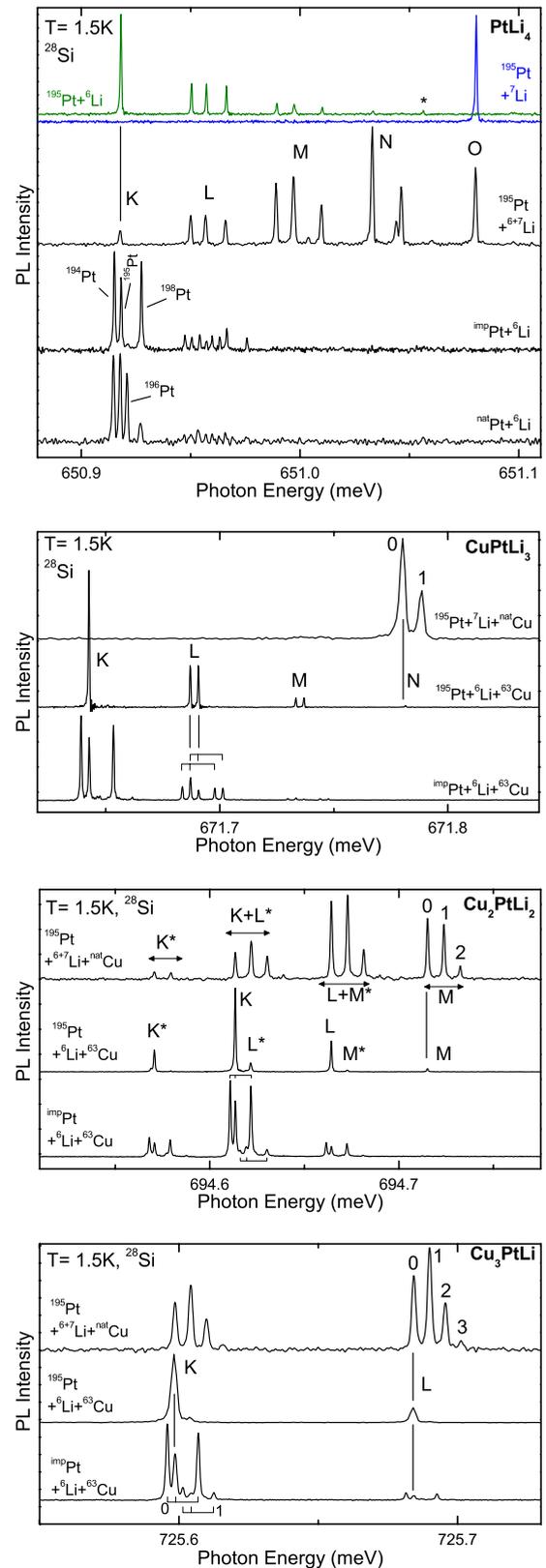


FIG. 5. (Color online) Spectra of the four-atom  $\text{Cu}_x\text{PtLi}_{(4-x)}$  series in  $^{28}\text{Si}$ . Numbers from 0 to 4 designate the number of  $^{65}\text{Cu}$  atoms in the cluster, letters K, L, M, N, and O designate the number of  $^7\text{Li}$  atoms in the cluster. Peaks labeled with a  $\star$  are of a different, overlapping electronic state. All spectra recorded at 1.5 K and resolutions better than  $1.4 \mu\text{eV}$ .

or more Pt atoms, there would be intermediate energy transitions from centers with mixed Pt isotopes. Hence we label it  $\text{Cu}_4\text{Pt}$ . It is clearly distinguishable from the 778 meV  $\text{Ag}_4$  complex.<sup>8</sup> Table I shows the energies of the two electronic states of  $\text{Cu}_4\text{Pt}$  for the isotopes  $^{63}\text{Cu}$  and  $^{65}\text{Cu}$ . The energies of all other peaks can be determined with the isotope shifts per atomic mass unit (amu) given in Table II. The much stronger PL from the higher energy state of  $\text{Cu}_4\text{Pt}$  at 776.926 meV (not shown) is not as well resolved as that of the weaker, lower energy transition which is shown in Fig. 2.

### B. $\text{Cu}_3\text{Pt}$ complex

The 884 meV  $\text{Cu}_3\text{Pt}$  system is shown in Fig. 3. Instead of five individual lines in the  $^{195}\text{Pt} + ^{\text{nat}}\text{Cu}$  spectrum this center has only four, labeled 0–3, showing the number of Cu atoms in the complex to be three. The two spectra with monoisotopic Cu clarify this labeling scheme. The two spectra at the bottom show the pattern that is characteristic for our Pt implanted and diffused samples. The natural Pt abundance is visible in the lower one, where the three lower energy peaks closely follow the expected relative intensities of  $^{\text{nat}}\text{Pt}$ . A residual amount of  $^{65}\text{Cu}$  remained visible in the  $^{63}\text{Cu}$  diffused sample, resulting in a slightly obscured spectrum, due to the overlap of the  $^{\text{nat}}\text{Pt}^{63}\text{Cu}_3$  (0) components with the weaker  $^{\text{nat}}\text{Pt}^{63}\text{Cu}_2^{65}\text{Cu}$  (1) components. We conclude that only one Pt atom is contained in this complex, which we thus label  $\text{Cu}_3\text{Pt}$ . The energies and isotope shifts of the three different  $\text{Cu}_3\text{Pt}$  electronic states are listed in Tables I and II.

### C. $(\text{Cu}, \text{Li}, \text{Pt})_4$ complexes

We now come to the newly discovered four-atom complexes related to  $\text{Cu}_3\text{Pt}$  but with Li substituting for Cu. Figure 4 shows the three centers,  $\text{Li}_3\text{Pt}$ ,  $\text{CuLi}_2\text{Pt}$ , and  $\text{Cu}_2\text{LiPt}$ . For all of them the spectrum of the Pt implanted sample shows the characteristic pattern already discussed for  $\text{Cu}_4\text{Pt}$  and  $\text{Cu}_3\text{Pt}$ . A spectrum of a sample with  $^{\text{nat}}\text{Pt}$  isotope abundances is only available for  $\text{Li}_3\text{Pt}$  due to the weakness of the PL in some of the samples. The relative intensities of 34%, 35%, 26%, and 5% of the PL lines of this spectrum accurately follow the natural isotope abundances of  $^{194}\text{Pt}$ ,  $^{195}\text{Pt}$ ,  $^{196}\text{Pt}$ , and  $^{198}\text{Pt}$ , respectively. Together this is evidence that there is only a single Pt atom in all four-atom Pt centers while the rest of the complex sites are filled with a combination of Li and Cu atoms. The figure also shows the isotope shifts due to different Cu and Li isotopes, where the labels K, L, M, and N stand for the number of  $^7\text{Li}$  atoms, 0, 1, 2, and 3, in the complex, in a scheme similar to the “0,” “1,” “2,” etc. labels used to denote the number of  $^{63}\text{Cu}$  in Cu-containing centers. It can thus be seen that these centers are of the form  $\text{Cu}_x\text{Li}_{(3-x)}\text{Pt}$ . Table I lists the energies of these centers and their different electronic states while Table II gives the isotope shifts per amu.

### D. $(\text{Cu}, \text{Pt}, \text{Li})_5$ complexes

As was observed for Au related complexes,<sup>10</sup> Pt also forms five-atom complexes together with Cu and Li in a

similar way. Figure 5 shows these PL centers that are labeled in a now familiar manner. For all four centers,  $\text{PtLi}_4$ ,  $\text{CuPtLi}_3$ ,  $\text{Cu}_2\text{PtLi}_2$ , and  $\text{Cu}_3\text{PtLi}$  a spectrum showing the characteristic pattern of our Pt implanted sample is given while in addition to that a spectrum of the  $^{\text{nat}}\text{Pt}$  diffused sample is given for  $\text{PtLi}_4$ . The relative intensities of 34%, 32%, 25%, and 9% of the PL lines of this center accurately follow the natural isotope abundances of  $^{194}\text{Pt}$ ,  $^{195}\text{Pt}$ ,  $^{196}\text{Pt}$ , and  $^{198}\text{Pt}$ , respectively. We can thus conclude that a single Pt atom is part of each of these centers. Additionally, spectra are displayed for each center to illustrate how Li is successively replaced by Cu from  $\text{PtLi}_4$  to  $\text{Cu}_4\text{Pt}$  (Fig. 2), following the scheme  $\text{Cu}_x\text{PtLi}_{(4-x)}$ . It should be noted that the electronic states of  $\text{PtLi}_4$  and  $\text{CuPtLi}_3$  shown in Fig. 5 display a substructure for the mixed Li configurations which was not observed for other electronic states of these two centers. Similar substructure has been observed before for  $\text{Cu}_4$  centers with mixed Cu isotopes,<sup>8</sup> and is likely due to the fact that in the present case, the Li sites must be inequivalent so that different arrangements of the same Li isotopes have different luminescence energies.

Directly after the Li diffusion the intensity of those four- and five-atom centers containing higher numbers of Li atoms is relatively low and the Cu-rich complexes are dominant. This ratio shifts in favor of the Li-rich centers when the samples are stored at room temperature for several weeks, indicating that Cu is slowly dissociated from the centers at room temperatures, and likely diffuses to the surface, being replaced by Li which remains at a high concentration within the sample due to its low diffusion coefficient at room temperature. Table I lists the energies of these centers and their different electronic states; the isotope shifts per amu can be found in Table II.

## IV. CONCLUSION

We have demonstrated that the method of isotopic fingerprints is a powerful tool to investigate deep photoluminescence centers containing Pt. Two of the discussed centers were previously known to be related to Pt. We could reinforce this previous result and determine the other constituents of these centers to be Cu, and we also showed that these centers contained a single Pt atom. In addition to this we have found series of four- and five-atom Pt complexes based on these centers with Li substituting for the Cu. Similar complexes have been observed for Au related deep level impurity centers and these four- and five-atom metal-related deep photoluminescence centers are now seen to be rather ubiquitous in Si.

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