

## Effect of surfactant Sb on carrier lifetime in GaInP epilayers

C. M. Fetzer, R. T. Lee, and G. B. Stringfellow<sup>a)</sup>  
*College of Engineering, University of Utah, Salt Lake City, Utah 84112*

X. Q. Liu, A. Sasaki, and N. Ohno  
*Academic Frontier Promotion Center, Osaka Electro-Communication University,  
 Neyagawa 572-8530, Japan*

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Samples of  $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$  grown on (001) GaAs with small amounts of surfactant Sb were investigated using time-resolved photoluminescence. All samples show a luminescence that may be fit to a two-stage exponential decay with a fast and a slow lifetime. For growth without Sb ( $\text{Sb}/\text{III}(v)=0$ ), the sample shows a strong  $\text{CuPt}_B$  ordering and a fast component lifetime of 7 ns. As the Sb concentration is increased, the degree of order is reduced, with a consequent increase in band gap energy. In the highest band gap material, produced at  $\text{Sb}/\text{III}(v)=0.016$ , the fast lifetime is 2.9 ns, an order of magnitude larger than published values for GaInP disordered by misorienting the substrate. Increasing the Sb further causes the band gap energy to decrease due to the onset of composition modulation. At  $\text{Sb}/\text{III}(v)=0.064$ , the fast component lifetime decreases to 0.79 ns. Samples grown with  $\text{Sb}/\text{III}(v)>0.016$  show a lifetime that depends on energy and is fit well by a model of localized excitons. © 2002 American Institute of Physics. [DOI: 10.1063/1.1423396]

### INTRODUCTION

Modern epitaxial methods such as organometallic vapor phase epitaxy (OMVPE) are capable of producing high-quality III–V semiconductor ternary alloys such as  $\text{Ga}_x\text{In}_{1-x}\text{P}$  (hereafter GaInP).<sup>1</sup> Typically, coherency strain from the substrate and the slow bulk diffusion kinetics limit phase separation in epilayers grown by this technique.<sup>1</sup> Adatom diffusion kinetics at the growth surface and surface thermodynamics lead to certain microstructures observed in these layers.<sup>2</sup> One prevalent microstructure is  $\text{CuPt}_B$  ordering, which forms due to the P dimers formed on the  $(2\times 4)$  reconstructed surface during growth.<sup>2,3</sup> In GaInP,  $\text{CuPt}_B$  ordering may be described as a monolayer superlattice of  $\text{Ga}_{(1+\eta)/2}\text{In}_{(1-\eta)/2}\text{P}/\text{Ga}_{(1-\eta)/2}\text{In}_{(1+\eta)/2}\text{P}$  along the  $[\bar{1}11]$  and  $[1\bar{1}1]$  directions, where the order parameter  $\eta$  describes the strength of the ordering ranging from 0 to 1.<sup>2–5</sup> For very rapid surface diffusion kinetics, another microstructure, composition modulation (CM), forms in GaInP.<sup>6</sup> CM is a self-assembled superlattice, which forms either lateral to the growth direction (lateral superlattice) or along the growth direction (vertical superlattice).<sup>6</sup> In GaInP, the lateral superlattice CM is typically observed as alternating In-rich and Ga-rich regions modulated along the  $[110]$  direction with a period of 10–60 nm.<sup>6</sup>

Since the surface thermodynamics and diffusion kinetics determine the final microstructure of the epitaxial layer, control of the surface allows the engineering of the final properties. A method recently introduced to control the surface is the use of small amounts of a surfactant.<sup>7</sup> A surfactant is an element that is neither readily incorporated into the solid nor easily evaporated from the surface. One such surfactant is

Sb. Sb has been observed in the growth of GaInP to: (i) eliminate  $\text{CuPt}_B$  ordering, (ii) induce triple period ordering, and (iii) cause the onset of composition modulation, all by selecting the amount of Sb on the surface.<sup>7–11</sup> Since Sb is isoelectronic with P, all of the effects occur without directly altering the bulk Fermi energy of the GaInP layer or by incorporating into the solid and forming an alloy.

$\text{CuPt}_B$  ordering and composition modulation strongly impact the optical properties of the epitaxial layer. Both microstructures produce a reduction in band gap energy from that of a completely random alloy.<sup>2,6,12</sup> Each microstructure also causes a strong optical polarization as observed by photoluminescence (PL).  $\text{CuPt}_B$  ordering produces stronger PL along the  $[110]$  direction. The polarization effect due to lateral superlattice CM is much stronger and oppositely oriented to that of  $\text{CuPt}_B$  ordering.<sup>6,13–16</sup> Thus, Sb may be used to selectively control the microstructure and hence the optical band gap and polarization.

The effects of Sb on the optical properties of GaInP have been only recently investigated.<sup>7,10,17</sup> One analysis has proposed that for certain Sb concentrations ( $\text{Sb}/\text{III}(v)>0.01$ ), the resulting luminescence is from excitons localized into potential minima of the CM structure.<sup>17</sup> The conclusions of that report were drawn from indirect evidence of power dependent PL.<sup>17</sup> A more direct method of determining the recombination mechanism is the use of time resolved PL (TRPL) to observe the luminescence decay behavior.<sup>18,19</sup> The purpose of this article is to examine the effect of Sb on OMVPE grown GaInP epilayers using TRPL methods.

### EXPERIMENT

Sample growth was performed in an atmospheric pressure, horizontal flow OMVPE reactor at 620 °C. Details of the epitaxy have been documented previously.<sup>7,9,10</sup> The epi-

<sup>a)</sup> Author to whom correspondence should be addressed; electronic mail: stringfellow@coe.utah.edu

layers are nominally grown on (001) GaAs substrates using conditions that produce strong  $\text{CuPt}_B$  ordering.<sup>7,9,10</sup> The only difference between samples was the introduction of various, small amounts of triethylantimony (TESb) during the growth.<sup>7,9,10</sup> The amount of Sb used in the process is characterized by the ratio of TESb to total group-III precursors in the vapor ( $\text{Sb/III}(v)$ ). Use of this ratio reflects the nonvolatile nature of the Sb, and correlates to the amount of Sb present on the surface of the layer during growth. Since the total V/III ratio in the vapor was held constant at 40, the results presented here may be compared to those of previous studies which were presented as  $\text{Sb/P}(v)$  simply by dividing by the V/III ratio.<sup>7,9</sup> A single sample grown on a (511)A GaAs substrate to produce disordered material without using Sb<sup>20</sup> was used as a control sample. Time independent PL (steady-state PL) measurements were made at 12 K using an Ar<sup>+</sup> laser operated at 488 nm with a power of 10 mW. All other details of the static PL are as described in previous studies.<sup>7,9,10,17</sup> PL decay measurements were made using a fast scan streak camera in conjunction with a 25 cm monochromator with a 100 lines/mm grating. Pulsed excitation was obtained from the mode-locked, frequency-doubled beam of an  $\text{Al}_2\text{O}_3\text{:Ti}$  laser at 400 nm pumped by a diode laser. The source repetition rate, nominally at 76 MHz, was selected to a value of 2 MHz, 400 kHz, or 80 kHz by means of an acoustic-optic modulator, with the lowest repetition rates used for the longer carrier lifetimes. The pulse width was 1.5 ps and the excitation power density was kept at  $1.4 \mu\text{J}/\text{cm}^2/\text{pulse}$ . The samples were held at 10 K during the measurements.

## RESULTS AND DISCUSSION

Figure 1, which shows the PL from three samples with increasing  $\text{Sb/III}(v)$ , summarizes the effects of Sb on the band gap energy and polarization of  $\text{GaInP}$ .<sup>10-17</sup> At  $\text{Sb/III}(v)=0$ , Fig. 1(a), shows the nominal behavior of  $\text{CuPt}_B$  ordered  $\text{GaInP}$ . The PL peak energy of 1838.2 meV indicates an order parameter of approximately 0.6.<sup>21</sup> The PL is also slightly polarized as shown by the inset figure of the PL intensity versus the angle of the polarizer. The PL along the  $[110]$  direction, at a  $\sim 45^\circ$  polarizer angle, is the strongest orientation. It is 1.66 times stronger than for the  $[\bar{1}10]$  direction, as would be expected.<sup>2,13-15</sup> Figure 1(b) shows the PL of the sample grown with  $\text{Sb/III}(v)=0.016$ . The PL peak energy has increased to 1958.7 meV as the ordering is decreased.<sup>7,8</sup> This sample has a PL peak energy 18.5 meV below that of the control sample grown on (511)A GaAs with a PL peak energy of 1977.2 meV (not shown). The polarization of the PL for this sample has dramatically increased. This result is counter to the weak polarization anticipated for the low ordering as determined by the high band gap energy.<sup>3,13-15</sup> Also, the orientation of the PL has reversed, with the  $[\bar{1}10]$  orientation now dominant, with an integrated intensity 21 times stronger than for the  $[110]$  polarization. The control sample grown without Sb demonstrated the expected behavior of weak polarization due to weak ordering, the  $[110]$  orientation being slightly stronger. Figure 1(c) shows that at  $\text{Sb/III}(v)=0.064$ , the PL peak en-

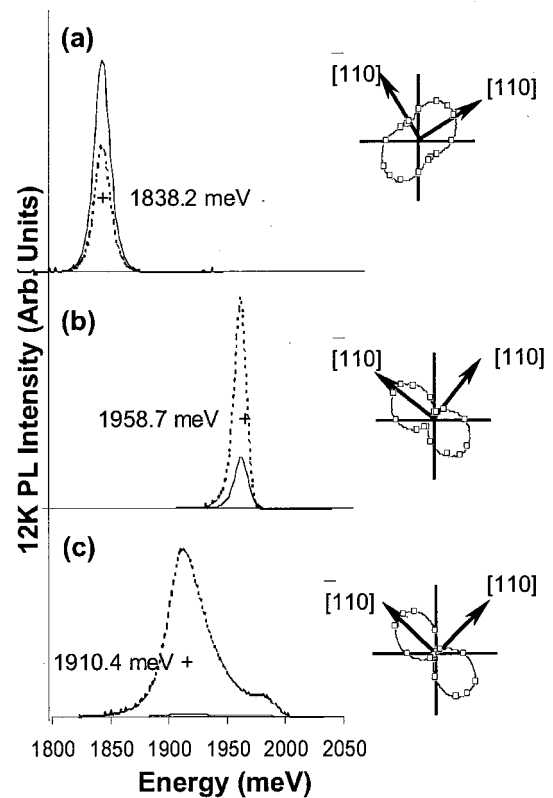


FIG. 1. PL intensity vs energy and polarizer angle for samples with: (a)  $\text{Sb/III}(v)=0$ , (b)  $\text{Sb/III}(v)=0.016$ , (c)  $\text{Sb/III}(v)=0.064$ . The 12 K PL spectra for each sample are shown on the left-hand side of the figure. The solid lines indicate a  $[110]$  polarizer orientation and the dashed lines indicate the  $[\bar{1}10]$  polarizer orientation. The inset figures on the right side show the peak PL intensity vs polarizer angle for each sample. The orientations of the  $[110]$  and  $[\bar{1}10]$  crystal axes relative to each figure are indicated.

ergy has decreased again to 1910.4 meV. Additionally, the polarization has dramatically increased such that the integrated PL intensity along the  $[\bar{1}10]$  orientation is more than 45 times stronger as is shown by the inset of Fig. 1(c). This band gap decrease was previously attributed to the presence of triple period ordering in this sample.<sup>9</sup> Recently, this hypothesis was demonstrated to be incorrect and the PL was shown to originate from a lateral superlattice CM present in both this sample and for all samples with  $\text{Sb/III}(v) > 0.01$ .<sup>17</sup>

Figure 2 shows the TRPL results for the samples from Fig. 1. All intensities are taken at a time from the initial pulse at the peak wavelength of the spectrum. The data are fit with a double exponential curve shown as the solid lines with a fast and slow component,  $\tau_{\text{fast}}$  and  $\tau_{\text{slow}}$ , respectively. The clear trend observed in the figure is that the lifetime decreases as  $\text{Sb/III}(v)$  increases. The sample with  $\text{Sb/III}(v)=0$  [Fig. 2(a)] shows lifetimes of  $\tau_{\text{fast}}=7$  ns and  $\tau_{\text{slow}}=29$  ns. These values are comparable to those published for moderately ordered  $\text{GaInP}$ .<sup>22</sup> The source of the slow component is speculative, but may be related to the indirect nature of recombination in ordered material.<sup>23-26</sup> The highest band gap material at  $\text{Sb/III}(v)=0.016$  shows lifetimes of 2.9 and 11.9 ns. At  $\text{Sb/III}(v)=0.064$ , Fig. 2(c) shows that the lifetimes decrease further to 0.79 and 3.74 ns for the fast and slow components, respectively. These lifetimes approach those for

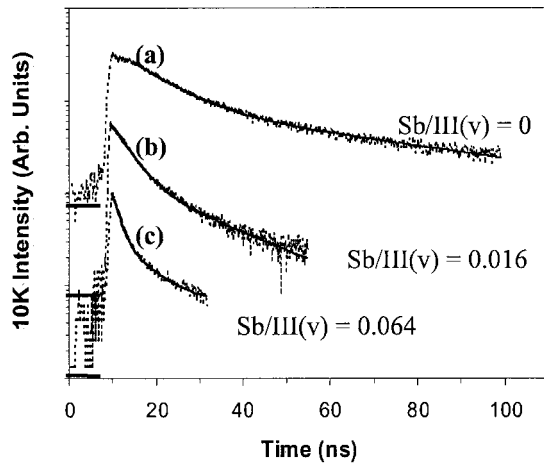


FIG. 2. 10 K PL intensity at the peak wavelength vs time for samples grown with: (a)  $Sb/III(v)=0$ , (b)  $Sb/III(v)=0.016$ , and (c)  $Sb/III(v)=0.064$ . Also shown is the two exponent curve fit (solid line) for each spectrum. Data are offset for clarity.

the control sample of 0.18 and 0.7 ns. It is clear that Sb has made an impact on the carrier lifetime as well as the PL peak energy in addition to the disordering observed previously.

The lifetimes are plotted versus  $Sb/III(v)$  in Fig. 3. The sample with the highest PL peak energy is marked by the arrow at  $Sb/III(v)=0.016$ . The general trend is again that the lifetime decreases as Sb is increased. Both the slow and fast components of the lifetime follow this trend, indicating that the recombination becomes more excitonic as the Sb concentration is increased. The effect is most likely due to the Sb on the surface during growth not the alloying of Sb. The concentration of Sb at the highest  $Sb/III(v)$  used in this study is  $\sim 10^{18} \text{ cm}^{-3}$ , as measured by (SIMS).<sup>17</sup> Since Sb has a lower electronegativity than P, one would expect the isoelectronic center to act as an electron trap.<sup>27</sup> All of the samples presented in this study are *n* type and hence the centers presumably would have no effect on the minority hole lifetime mea-

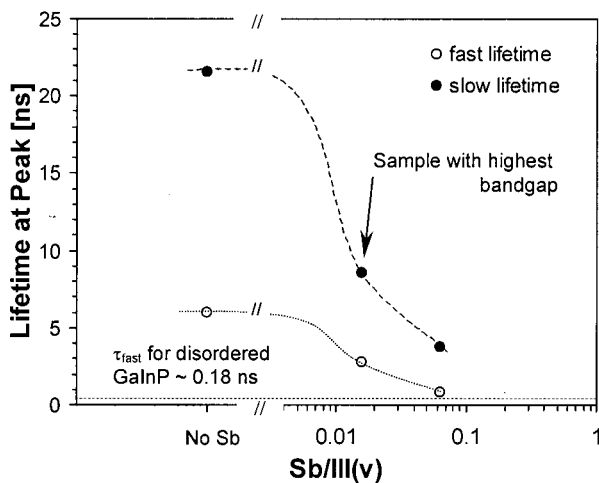


FIG. 3. Lifetime from curve fit vs  $Sb/III(v)$ . The fast (slow) lifetimes are indicated by the open (filled) circles. The fast lifetime of the control sample is shown as the horizontal dashed line at 0.18 ns. The sample grown with an Sb concentration ( $Sb/III(v)$ ) of 0.016 giving the highest band gap is indicated by the arrow.

sured here.<sup>17</sup> Additionally, isoelectronic centers in III-V materials have very weak binding, typically less than the 9 meV observed for N centers in Gap, or act as deep levels.<sup>28</sup> All of these points indicate that the low concentrations of Sb in the solid have a negligible direct effect on the observed lifetime behavior. Another point to consider is that the most disordered sample ( $Sb/III(v)=0.016$ ) shows lifetimes 1 order of magnitude longer than those of the control sample. Both samples have nearly the same band gap and PL full width at half maximum. The use of Sb to control the ordering has lengthened the minority carrier lifetime by 1 order of magnitude over that observed in a sample disordered by substrate misorientation. This result demonstrates a possible advantage of using Sb to control ordering in device manufacture where a long lifetime is required. Further investigation into this observation may be warranted.

The trend to faster lifetimes with increased  $Sb/III(v)$  seen in Fig. 3 is consistent with the presence of CM in the samples. Above  $Sb/III(v)>0.016$  the lifetimes are comparable to those from published TRPL studies of GaInP with CM, where the fast component of lifetime is reported to be 1 ns.<sup>18,19</sup> As proposed in earlier studies,<sup>17</sup> the luminescence from the sample grown with  $Sb/III(v)>0.016$  is from recombination localized in the low band gap areas in the lateral superlattice. Even at  $Sb/III(v)=0.016$ , the localization is enough to significantly alter the lifetime of the excitons as compared to that of the control sample.

A further test of the localized exciton hypothesis in the samples grown with  $Sb/III(v)>0.016$  is that lifetimes should follow the exciton transfer model.<sup>18,19,29</sup> Such a model has been previously used to describe the lifetimes of GaInP epilayers with CM present.<sup>18,19</sup> In the exciton transfer model, excitons may transfer from a high energy valley into a region of lower energy before recombining.<sup>29</sup> Two competing processes exist for a given exciton, and transfer to another localized state with a lower energy and radiative recombination. The competition of the two processes, transfer and recombination, has been modeled assuming an exponential tail in the density of exciton states described by  $\rho(E) = \rho_0 \exp(-E/E_0)$ , where  $E_0$  describes the amount of spreading in the density of states  $\rho$ .<sup>29</sup> This same functional form describes the averaged density of states of the lowest lying band gap regions with random fluctuations. In such a case, the observed lifetime increases with decreasing energy, becoming equal to the recombination lifetime at lower energy as fewer lower energy states exist to which the exciton may transfer.<sup>29</sup> The relationship between lifetime  $\tau(E)$  and PL energy  $E$  is given by

$$\tau(E) = \frac{\tau_0}{1 + \exp\left(\frac{E - E_{cm}}{E_0}\right)}, \quad (1)$$

where  $\tau_0$  is the lifetime of the exciton recombination and  $E_{cm}$  is the energy of the “mobility” edge. In the CM structure, this energy is the average energy of the well minima.

The fast component of lifetime is plotted versus energy in Fig. 4 for both samples for  $Sb/III(v)=0.016$  [Fig. 4(a)] and  $Sb/III(v)=0.064$  [Fig. 4(b)]. The solid line is a least

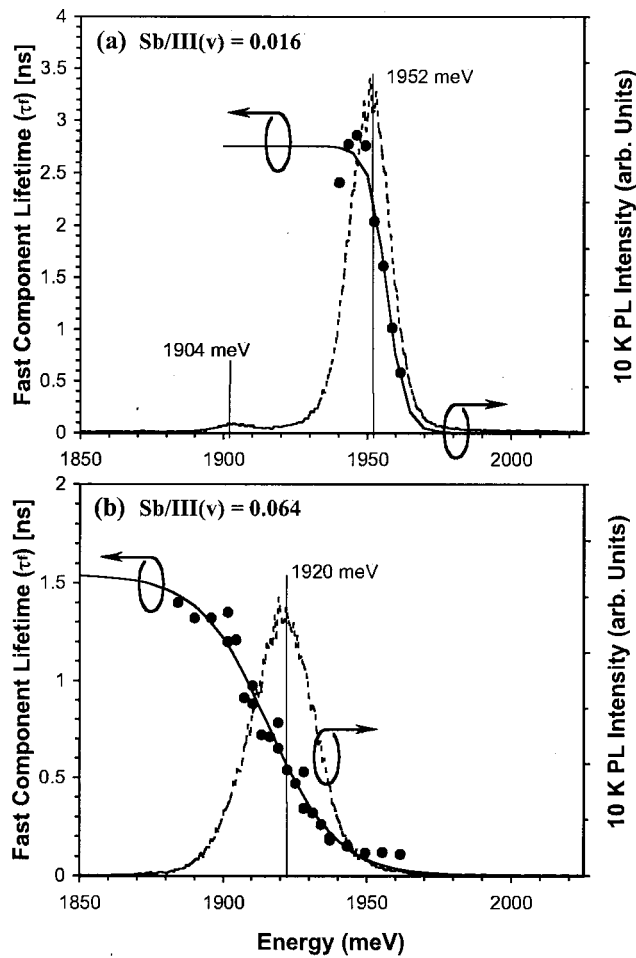


FIG. 4. Fast component lifetime vs wavelength for samples with (a)  $\text{Sb/III}(v) = 0.016$  and (b)  $\text{Sb/III}(v) = 0.064$ . Dashed lines indicate the 10 K PL spectra at maximum intensity and correspond to the right-hand axis. The solid line is a fit to the equation from the localized exciton model.

squares fit of Eq. (1) to the data. Static 10 K PL spectra are also shown as dashed lines. The peak at 1902.3 meV in the spectrum for  $\text{Sb/III}(v) = 0.016$  is easily identified as a phonon replica of the main peak.<sup>30</sup> Results of fitting the lifetime to Eq. (1) for all samples are given in Table I. The validity of the model was estimated from the  $\chi^2$  test of the fit equation to the data. A  $\chi^2$  value of 0 indicates a perfect fit of all data to the equation.<sup>31</sup> The data were fit with an automated routine to minimize  $\chi^2$ . The control sample shows the best fit to the equation. The poorest fit is shown by the  $\text{CuPt}_B$  ordered sample. The result indicates that the exciton transfer model is not applicable to the  $\text{CuPt}_B$  ordering. This may be due to the indirect recombination reported for  $\text{CuPt}_B$  ordering.<sup>23–26</sup>

Both samples grown with  $\text{Sb/III}(v) > 0.016$  show a good fit that improves with increased Sb in the vapor. The fraction of carriers recombining via localized excitons increases with the amount of Sb. The result correlates to the observed increase in CM amplitude with increased  $\text{Sb/III}(v)$ .<sup>17</sup> As the Sb is increased the modulation amplitude also increases, resulting in further localization of the excitons in the potential minima. In both samples, the mobility edge has the same value as the PL peak energy. In terms of CM, minima of the low band gap regions are centered in energy about the PL peak energy value. This contrasts directly with the samples grown without Sb where the mobility edge is calculated to be about 20 meV above the observed PL peak energy.

The spread in density of states ( $E_0$ ) is also affected by the Sb concentration. With the exception of the undoped sample,  $E_0$  appears to decrease at  $\text{Sb/III}(v) = 0.016$  from that of the control sample and then increase again as the  $\text{Sb/III}(v)$  increased. The larger  $E_0$  indicates a greater fluctuation in the bottom of the wells in the CM structure as the Sb concentration is increased. In other words, the nonuniformity of the CM increases with the amplitude of the composition modulation.

Finally, the exciton recombination lifetime  $\tau_0$  follows the same trend with increasing Sb concentration as described previously for the fast and slow component lifetimes. It decreases with increased Sb concentration, and it is a full order of magnitude longer for the most disordered sample at  $\text{Sb/III}(v) = 0.016$  than for the control sample.

## SUMMARY

In summary, addition of Sb during OMVPE growth of GaInP has been shown to decrease the minority carrier lifetime. A monotonic decrease in  $\tau$  with increased Sb is observed. For  $\text{Sb/III}(v) > 0.016$ , a strong excitonic component of the luminescence is present that becomes more dominant as Sb is increased. At the maximum disordering due to Sb, lifetimes are 1 order of magnitude longer than for a comparable control sample. An excellent fit to the exciton transfer model is obtained at high Sb concentrations. The results indicate that for samples grown with  $\text{Sb/III}(v) > 0.016$ , the PL originates from localized excitons in a CM structure.

## ACKNOWLEDGMENTS

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TABLE I. Least-squares fitting results for localized exciton model.

Sample	$\text{Sb/III}(v)$	PL peak (meV)	$E_{cm}$ (meV)	$\tau_0$ (ns)	$E_0$ (meV)	$\chi^2$	Microstructure observed
Control (Sb-28a)	0.0	1977.2	1992	0.24	8.2	0.025	Disordered-none
Sb-12	0.0	1838.2	1859	5.6	4.8	7.389	$\text{CuPt}_B$ ordering
Sb-20	0.016	1958.7	1955	3.11	5.0	0.838	CM
Sb-19	0.064	1910.4	1916	1.54	11.9	0.442	CM

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