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Study of the incorporation factor (K) and its application to laser diode production

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Abstract

A distribution coefficient, K, can be used to simply and effectively track how species distribute between the gas phase and solid phase. Alfalight, Inc., a manufacturer of high-power laser diodes, uses this coefficient in several aspects of epitaxial growth for laser diode production. First, Alfalight, Inc. uses K over different materials and across a wide range of material compositions within a specific material system to study the incorporation dependencies of species as a function of growth conditions. K as a function of material composition will be discussed with special attention paid to the $In_xGa_yAl_{1-x-y}P$ materials system. $In_xGa_yAl_{1-x-y}P$ is particularly interesting because the Al incorporation factor increases with increasing Al composition in the epitaxial layer. In addition, the K factor methodology lends itself to the use of statistical process control (SPC) methods. Alfalight, Inc. has used SPC to prove that the MOVPE process can be considered to be an in-control process-a significant benefit to manufacturability. The use of the K factor methodology as an SPC tool will be discussed with specific examples. The major advantage of the K-factor methodology is that it allows control of the growth of the wide composition range of III–V semiconductor layers that Alfalight, Inc. uses to manufacture multi-mode broad area lasers (MMBAL) and single mode lasers at 808, 915, and 980 nm emission wavelengths, for example.

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1. Introduction

The application of statistical process control (SPC) strategy to laser diode production is a necessary step toward manufacturability [1]. The goal of the SPC strategy is to monitor the variation in the process and identify and eliminate uncontrolled variation so that a process is operating within its natural process window. The first step in applying SPC strategy to a metal organic vapor phase epitaxy reactor is the selection of the variables that are to be used in the control charts.

An obvious choice for many devices is the use of photoluminescence wavelength and X-ray lattice mismatch. These variables are reasonable selections because they are directly measured values of materials properties. Alfalight Inc., a manufacturer of high-power laser diodes, is instead investigating the use of a distribution coefficient or K_{factor} as an SPC tracking value.

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The K_{factor} is a number that can be applied to both group III and group V species and simply describes how species distribute between the gas and solid phases [2]. The incorporation of gas-phase species into the solid is a complicated process that may involve both thermodynamic and kinetic phenomena. Regardless of the driving force behind the incorporation, each species will incorporate into the solid based on some conversion factor from the gas phase. For example, in the growth of $In_xGa_{1-x}P$ the composition of In will be expected to follow the relationship:

$$X_{In} = \frac{\Theta_{In}*F_{In}}{\Theta_{In}*F_{In} + \Theta_{Ga}*F_{Ga}}, \tag{1}$$

where F_i and Θ_i indicate the molar flow and incorporation factor, respectively. The K_{factor} is derived by arbitrarily assigning one species unity incorporation. For these investigations Ga will be assigned unity incorporation since Ga is a constituent of every material grown at Alfalight, Inc.

$$X_{In} = \frac{\Theta_{In}*F_{In}}{\Theta_{In}*F_{In} + \Theta_{Ga}*F_{Ga}} = \frac{(\Theta_{In}/\Theta_{Ga})*F_{In}}{(\Theta_{In}/\Theta_{Ga})*F_{In} + F_{Ga}} = \frac{K_{In}*F_{In}}{K_{In}*F_{In} + F_{Ga}}. \tag{2}$$

A similar derivation can be employed for mixed group V components, $In_xGa_{1-x}As_yP_{1-y}$ for example, where As is typically assigned unity incorporation relative to P.

Control-charting strategies are essential to manufacturing operations using MOVPE reactors independent of the variables that are tracked. First, the application of any control chart provides insight into the state of the MOVPE reactor. The identification and elimination of uncontrolled variation reduces the necessity of "tweaking" the MOVPE growth process typically performed to meet material specifications, in favor of identifying the natural process window [1].

The choice of the K_{factor} as a control chart variable provides several interesting advantages. The selection of the K_{factor} for the control chart variable provides data for rapid development of a semi-empirical model for MOVPE growth. This model allows for straightforward adjustment of flow rates in order to predict new flows for new material compositions. This has the benefit of reducing the number of required calibrations to develop a new materials set. While a complete thermodynamic and kinetic model would be the best choice, economic factors may prevent such a rigorous undertaking. Therefore, compromising with the development of a semi-empirical model provides practical utility without undue economic burden.

In addition, the K_{factor} methodology can reduce the calibration sequence for device epitaxy, resulting in a cost reduction. A typical calibration process would involve identifying the material set and calibrating each material within the desired device structure. This procedure may result in as a many as five or six layer calibrations, depending on the specific device type. However, the application of a K_{factor} approach may only require one or two calibrations depending on the material set. Once a database of existing K_{factor} has been established and the relationships identified, future calibrations could be reduced. The procedure would involve verifying that the K_{factor}s for the species, say In and P, are within normal process limits and then the device growth can be performed with confidence. Judicious selection of the calibration layer would be required in order to have confidence in each K_{factor} required for the device.

The K_{factor} also yields itself to a more intuitive understanding of reactor deviations. For example, it is readily understood that K_{In} decreases with increasing reactor temperature [3]. However, photoluminescence wavelength, another natural control chart variable, can be affected not only by In composition variations, but also by the degree of ordering, which changes with growth parameters such as temperature, growth rate, and V/III ratio, for example [4,5]. Therefore, care must be taken when interpreting changes in photoluminescence wavelength that are not necessary when tracking K_{In}.

The remainder of this article will focus on specific applications of the K_{factor} approach used at Alfalight in the manufacturing of high-power laser diodes over a wide range of material compositions.

2. Experimental details

The MOVPE growths were performed at reduced pressure in a close-coupled showerhead (CCS) vertical reactor. Epitaxial layers of $Al_xGa_{1-x}As$, $In_xGa_yAl_{1-x-y}P$, $In_xGa_{1-x}As_yP_{1-y}$, and $In_xGa_{1-x}P$ were grown lattice matched to GaAs in the temperature range 630–750°C. The group III precursors were trimethylgallium (TMG), trimethylindium (TMI), trimethylaluminum (TMA) while the group V precursors were AsH_3 and PH_3. The $Al_xGa_{1-x}As$ and $In_xGa_{1-x}P$ compositions were determined from resulting film composition was determined using double crystal X-ray diffraction on a commercially available system. $In_xGa_{1-x}As_yP_{1-y}$ and $Al_xIn_yGa_{1-x-y}P$ film compositions were determined using room temperature photoluminescence and double crystal X-ray diffraction on compositions where ordering does not occur. The K_{factor} for the ith component was calculated based on gas-phase delivery to the reactor, measured film composition and rearrangement of Eq. (2) to yield:

$$K_i = \frac{F_{\mathrm{Ga}}}{F_i * (1/x_i - 1)}. \tag{3}$$

3. Results and discussion

The goals of the remainder of this article are to detail how Alfalight, Inc. has used the K_{factor} in the SPC charting and how that has impacted the device epitaxy at Alfalight. To that end, specific examples have been selected to highlight the K_{factor} utility.

3.1. Demonstration of K_{Al} for $Al_xGa_{1-x}As$ growth

The first goal of a control chart strategy is to identify the state of process. The ideal state is a process that is inherently stable over time, operating within the natural process window [1]. Fig. 1 provides a control chart of K_{Al} for over 100 epitaxial growths of $Al_{0.55}Ga_{0.45}As$.

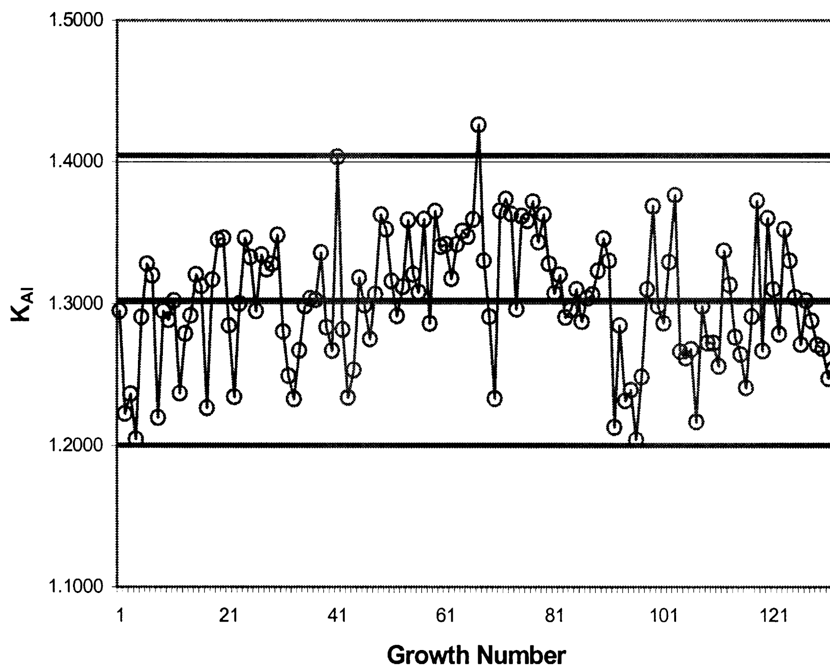

Fig. 1. K_{Al} for $Al_{0.55}Ga_{0.455}As$ growth. Growth conditions for these 100 + runs are identical. The lower, middle, and upper lines indicate the lower process limit (LPL), process average, and upper process limit (UPL), respectively.

Immediately, it was recognized that the growth of $Al_xGa_{1-x}As$ was a textbook example of an in-control process. This exercise provided confidence that the K_{Al} value was 1.3 ± 0.1, translating practically to a $\pm 2\%$ Al natural process window. In other words, when targeting 55% Al, any composition from 53% to 57% Al would be considered within the natural process variation and should not result in a "tweak" to the growth conditions. The fact that the K_{Al} value is in control suggests the possibility of reducing the required number of calibrations for this material set and instead maintaining a control chart that simply flags when the growth falls out of control. This provides a potential for reduced cost in manufacturing operations using $Al_xGa_{1-x}As$.

An important advantage of the K_{factor} approach is the ability to generate a semi-empirical model for predicting changes in flows required to change material compositions. A database of K_{factor}s as a function of doping and temperature provides a simple way to predict the required flows for a new material composition. This reduces the number of calibrations and leads to cost savings when attempting to calibrate new material compositions. However, care must be taken when changing materials compositions because as mentioned earlier, the K_{factor} can change as a function of composition. The most obvious examples of this are the K_{Al}s for $In_xGa_yAl_{1-x-y}P$ and $Al_xGa_{1-x}As$ growth as shown in Figs. 2 and 3.

Clearly K_{Al} is a function of the Al concentration required in the solid. This composition dependence, although not large, may indicate a phenomenon that occurs in the CCS that is not typically seen in all reactor geometries and requires further investigation. The origin of this composition dependence is not completely understood at this time. One potential theory could relate to the increased bond strength of the Al atom relative to the other species [6]. Perhaps introducing more Al into the chamber results in an increased incorporation of the Al species due to a stronger binding of the Al atoms to the growing surface. Or perhaps increased surface competition between Ga and Al absorbed species leads the Al to incorporate more quickly. Interestingly, as shown in Fig. 3, the K_{In} value is not impacted by the changing K_{Al} value.

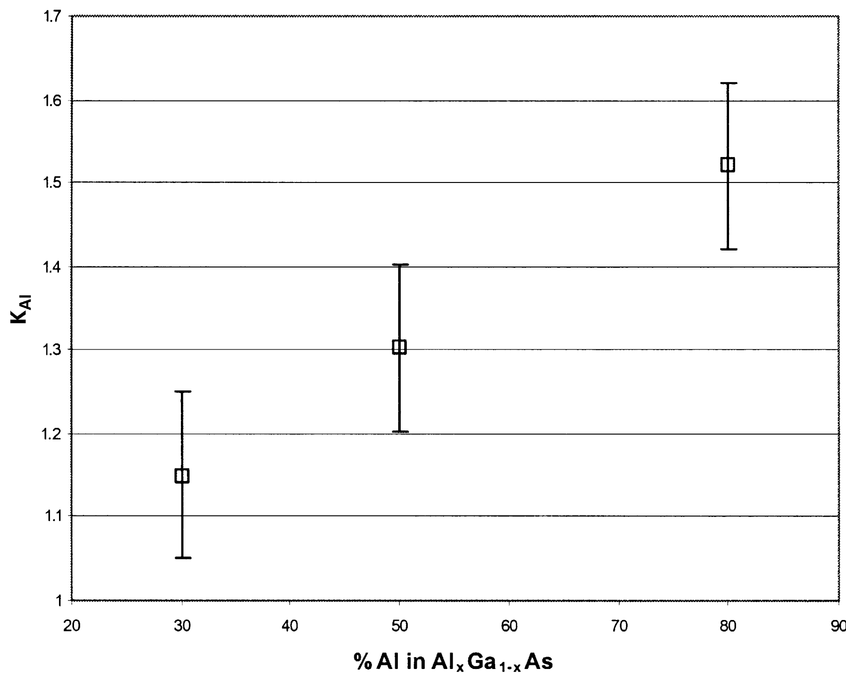

Fig. 2. K_{Al} for $Al_xGa_{1-x}As$ growth as a function of Al composition.

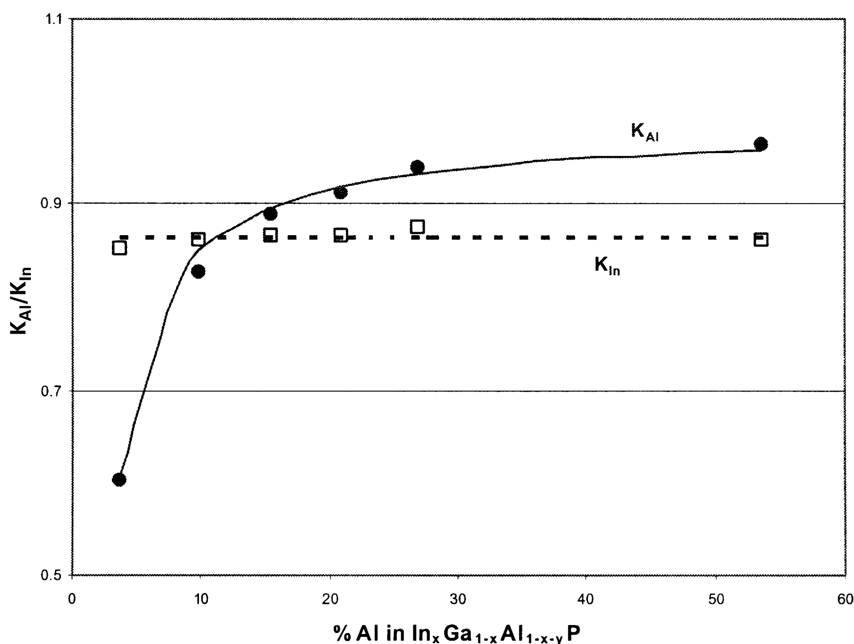

Fig. 3. K_{Al} (—●—)and K_{In} (—□—) for $In_xGa_yAl_{1-x-y}P$ growth. K_{In} is independent of composition while K_{Al} increases with increasing Al incorporated into the solid.

Further investigations will be required to understand this result more completely. Regardless of the physical explanation, this data indicates that changing Al composition via this semi-empirical model requires some understanding of the K_{factor} dependence on material composition.

3.2. K_{In} for $In_xGa_{1-x}As_yP_{1-y}/In_xGa_{1-x}P$ growth

K_{In} has also been used in control charting at Alfalight, Inc. Figs. 4 and 5 provide control charts of K_{In} for un-intentionally doped uid-$In_xGa_{1-x}As_yP_{1-y}/In_xGa_{1-x}P$ as well as uid- and p-type $In_xGa_{1-x}P$, respectively. Again, the K_{In} value demonstrates an in-control process. Fig. 4 clearly indicates that the K_{In} is dependent on the material composition, which is an expected result compared with modeling data [7]. Previous reports have shown that the group III incorporation is dependent on the group III concentrations due to different kinetic factors of the group III species [7]. A particularly interesting observation is that knowing that the K_{In} value is in-control for one material system implies control for the other material system. Therefore, it is no longer necessary to calibrate both $In_xGa_{1-x}As_yP_{1-y}$ and $In_xGa_{1-x}P$ materials prior to device growth.

The effect of Zn doping on the offset in K_{In} is also an expected result since In and Zn are known to have an interaction in MOVPE growth [8]. The impact of Zn on the K_{In} value provides a means of ensuring the correct Zn doping level prior to detailed electrical characterization. Investigations at Alfalight, Inc. have shown a one-to-one correlation between the value of K_{In} and the measured sheet resistance of the doped layer up to $1e18\,cm^{-3}$ Zn doping. Therefore, knowledge of the K_{In} value obtained on p-$In_xGa_{1-x}P$ layers also indicates a Zn doping level that can be confirmed with detailed electrical measurements at a later time. This is particularly useful for device layers with multiple doped layers where characterization is difficult by straightforward van der Pauw Hall or sheet resistivity measurements. The K_{In} value can be readily obtained from device growths, which ensures that the dopant level is correct and

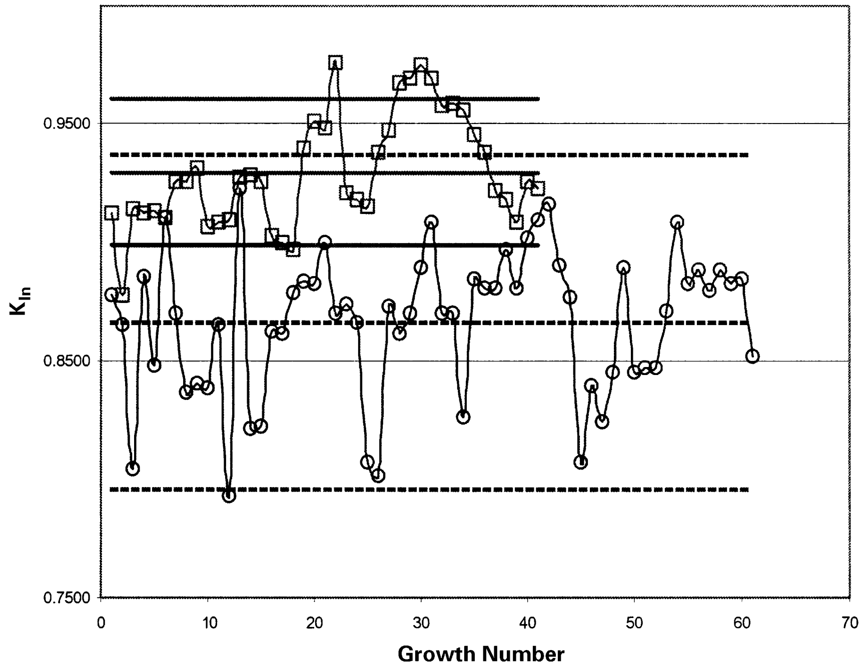

Fig. 4. K_{In} for $\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}$ (—□—) and $\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{AsyP}_{1-y}$ ($E_g = 1.62\,\mathrm{eV}$) (—○—) at the same growth temperature. For each composition the lower, middle, and upper lines indicate the LPL, process average, and UPL, respectively.

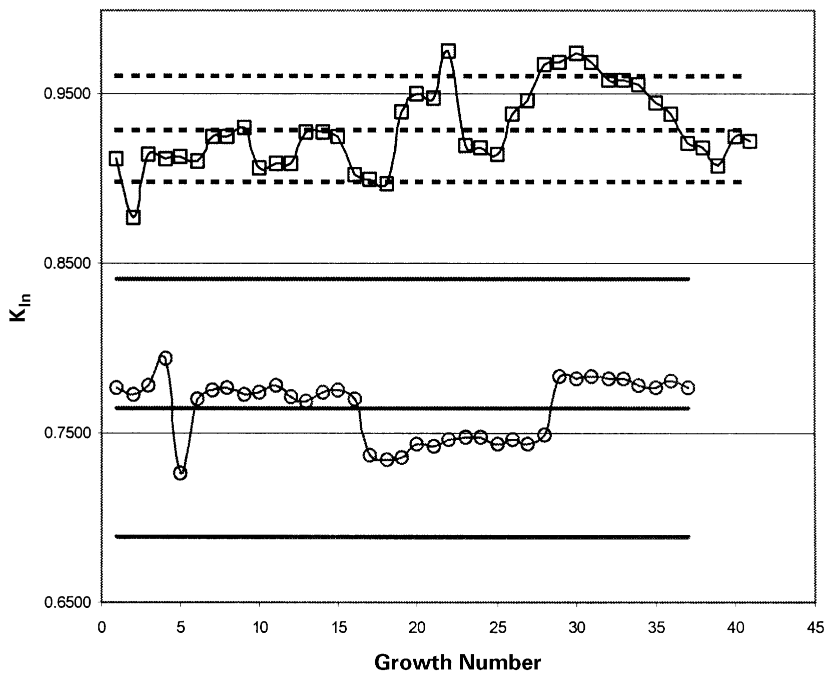

Fig. 5. K_{In} for uid-$\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}$ (—□—) and p-$\mathrm{In}_x\mathrm{Ga}_{1-x}\mathrm{P}$ (c.c. = 1e18 cm^{-3}) (—○—) at the same growth temperature. For each doping level the lower, middle, and upper lines indicate the LPL, process average, and UPL, respectively.

lessens mistakes of growing p-type layers with an empty or near empty dopant cylinder.

3.3. K_p control chart in a trouble shooting application

One noted advantage of the K_{factor} methodology mentioned previously is the intuitive nature of the variable for interpreting reactor status (e.g. temperature stability, pressure control, MFC control). This has been used at Alfalight, Inc. during troubleshooting scenarios. For example, at Alfalight $In_xGa_{1-x}As_yP_{1-y}$ with $E_g = 1.62\,eV$ and 0 PPM strain is a standard material. The K_P control chart for $In_xGa_{1-x}As_yP_{1-y}$ ($E_g = 1.62\,eV$) layers is provided in Fig. 6.

Inspection of Fig. 6 shows a decreasing trend in K_P with time. In fact, this process is considered out-of-control when using rigorous control rules [1]. In addition, the upper and lower process limits associated with this control chart indicate a large variation in the As mole fraction of the resulting solid for a given AsH_3 flow. Clearly, this variation would not result in an acceptable material composition variation. A maintenance activity was triggered as a result of this out-of-control process. Intuitively, K_P decreases with decreasing temperature [9,10]. Therefore, trouble-shooting activities focused on temperature measurements. Early in the maintenance activity, a failing thermocouple responsible for monitoring the reactor temperature was found and replaced. This eliminated the variation in K_P and restored the value to the original measurement. Therefore, the K_{factor} methodology provided an intuitive means of troubleshooting a control problem and minimized overall reactor downtime.

4. Conclusions and recommendations

The K_{factor} has been employed at Alfalight, Inc. as a control-charting variable. While other variables such as photoluminescence wavelength and X-ray lattice mismatch would provide useful control charts, the K_{factor} may be better for a

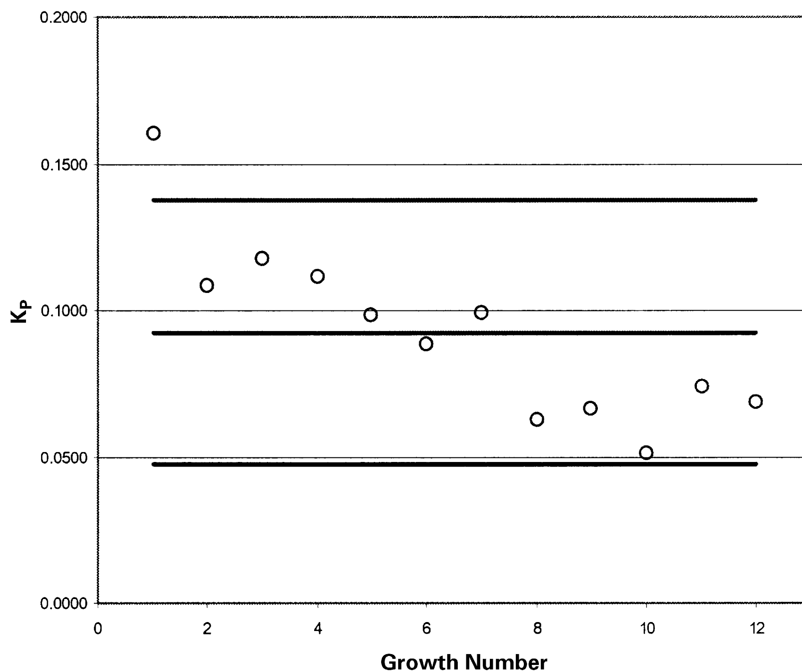

Fig. 6. K_P control chart for $In_xGa_{1-x}As_yP_{1-y}$ ($E_g = 1.62\,eV$). The lower, middle, and upper lines indicate the LPL (47.7% P), process average (57.5% P), and UPL (72.7% P), respectively.

number of reasons. First, the database of K_{factor}s developed in the control charting strategy allows for development of a semi-empirical model of the MOVPE growth process allowing quick prediction of new flows for new materials in laser diode product development. Second, the relationships discovered between K_{factor}s for different material compositions translate into a reduced number of required calibrations prior to device growth. For example, knowing that the K_{In} for $In_xGa_{1-x}As_yP_{1-y}$ ($E_g = 1.62\,eV$) is in control readily correlates to the K_{In} for $In_xGa_{1-x}P$ being in control, leading to the conclusion that the MOVPE system is operating within natural process variation. Finally, the K_{factor} provides an intuitive variable for understanding the state of the reactor. The K_{In} value can be directly correlated to the layer doping level for Zn-doping providing knowledge of the doping level without detailed electrical characterization or destructive testing of device structures. Alfalight, Inc. has employed this methodology in the production of multi-mode broad area lasers (MMBALs) at wavelengths of 808, 915, and 975 nm. This flexible and effective variable for developing control charts has provided a means of calibrating materials systems across all of these wavelengths yielding excellent device performance as shown in Table 1. This excellent laser diode performance indicates high-quality epitaxial growth that has been facilitated by the use of the K_{factor} as a control chart variable.

Table 1
Performance characteristics for Alfalight, Inc high-power laser diodes

Device type	SE (W/A)	Loss (cm^{-1})	Life time
975MMBAL	>0.95	<1.0	ML = 75 year for 2W at 25°C
915MMBAL	>1.0	<1.0	Measurement in progress
808MMBAL	>1.1	$<1/5$	Measurement in progress

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