Fluorocarbon impurities in KrF lasers

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Fluorocarbon impurities are known to have deleterious effects on the operation of excimer lasers; however, the sensitivity limits are poorly known. Absorption at 248.9 nm in an e-beam-pumped KrF laser has been attributed to CF_2, produced by plasma fragmentation of precursor molecules such as CF_4. In this paper, the effects of CF_4 impurities on the gain of an electron-beam-excited KrF laser are investigated theoretically. It is found that the density of $KrF(B)$ significantly decreases and absorption increases when the CF_4 concentration exceeds 0.03%. The decrease in the density of $KrF(B)$ is dominantly the result of the interception of precursors to forming the upper laser level, as opposed to direct quenching.

I. INTRODUCTION

The performance of electron-beam- (e-beam) and discharge-excited excimer lasers is known to be sensitive to trace impurities.[1] These contaminants are present in the feedstock gases, are generated by plasma decomposition of other innocuous impurities or by the decomposition of the feedstock gases themselves.[2,3] Laser efficiency was measured by Kimura and Seamans in an e-beam-excited XeF laser during controlled impurity addition.[4] They observed a monotonic and nearly linear decrease in laser efficiency upon addition of CF_4, O_2, HF, CO_2, CO, and SiH_4. An addition of 0.1% CO was sufficient to extinguish the laser while an addition of the same amount of CF_4 decreased laser efficiency by 25%–30%. Mandl and Hyman attributed the superior performance of repetitively pulsed XeF lasers in mixtures using F_2 compared to NF_3 to buildup of N_2 by plasma decomposition of NF_3.[2] They hypothesized that charge exchange of N_2 with Ne_2^+ forming N_2^+ $(X,v = 3,4)$ resulted in absorption at the laser wavelength ($\lambda = 351$ nm).

ArF and KrF excimer lasers also have shown unexpected sensitivity of small amounts of impurities, and fluorocarbons in particular.[1] Fluorocarbons such as CF_4 are common contaminants in the feedstock halogen gases (most often F_2), or are generated *in situ* from reactions of fluorine with organic contaminants resulting from sealants, solvents, or pump oils. The origin of the sensitivity of e-beam-excited KrF lasers to this contamination is not clear. It is known that reactions of Kr* with CF_4 molecules most often quench Kr* as opposed to resulting in the harpooning reaction leading to excitation of the upper laser level as occurs with F_2.[5] Therefore, interception of precursors to the upper laser level (i.e., Kr*, Kr_2^*) by CF_4 could result in a decrease in laser performance. CF_4 is also an attaching gas which forms the negative ion fragments F^- and CF_3^-.[6] Ion–ion neutralization reactions of Kr^+ and Kr_2^+ with F^- result in excitation of the upper laser level $KrF(B)$.[7] Similar reactions of Kr^+ and Kr_2^+ with

CF_3^- may not populate $KrF(B)$ and therefore may be intercepting process that reduce the density of precursors to the upper laser level. CF_4 impurities can, of course, directly quench the upper laser level as well.

CF_4, as well as other fluorocarbon impurities, are not absorbing at the $KrF(B \rightarrow X)$ laser wavelength ($\lambda = 248.4$ nm).[8] Plasma decomposition products of the impurities, though, may be absorbing. Therefore spectroscopic analysis of the unexcited gases may not be a good indication of the absorbing potential of the impurities. Absorption at 248.9 nm in the laser spectrum of KrF has been attributed to the $CF_2(X \rightarrow A)$ transition.[9,10] The cross section for this process has been estimated to be 2×10^{-17} cm^2.[9] Therefore significant absorption ($>0.05\%$ cm^{-1}) occurs with CF_2 concentrations of $2–3 \times 10^{13}$ cm^{-3}, or ≈ 1 ppm in atmospheric pressure mixtures.

In this paper, we report on the results of a theoretical investigation of the consequences of contamination of electron-beam-excited KrF lasers by CF_4. This study was performed using a computer model for the plasma chemistry in e-beam-excited laser in $Ar/Kr/F_2$ mixtures. We find that the impurity level of CF_4 at which laser performance is significantly degraded is approximately 0.03%–0.01%, or 300–1000 ppm, and that the degradation is a consequence of both increased quenching of precursors to the upper laser level and increased absorption.

II. DESCRIPTION OF THE MODEL

The model used in this study is based on a previously described simulation for an e-beam-excited KrF laser.[11] The model accounts for the slowing of an injected e-beam in a high-pressure plasma, and the resulting electron kinetics, heavy particle reactions, and photophysics. The kinetics used in this work for pristine $Ar/Kr/F_2$ mixtures differ little from that described in detail by Kannari, Obara, and Fujioka[12] and Czuchlewski et al.,[13] and the reader is referred to those works for a discussion of the plasma chemistry of clean systems. Electron-impact rate coefficients and

TABLE I. Fluorocarbon reactions included in the model.

Reaction	Rate coefficient[a]	Reference
$e + CF_4 \rightarrow CF_3^+ + F + e + e$	7.60(− 14)	b,16,25
$e + CF_4 \rightarrow CF_3 + F + e$	6.30(− 14)	b,15,16
$e + CF_4 \rightarrow CF_2 + F + F + e$	1.47(− 13)	b,15,16
$e + CF_4 \rightarrow CF_3 + F^-$	1.06(− 12)	b,6,16
$e + CF_4 \rightarrow CF_3^- + F$	4.24(− 12)	b,6,16
$e + C_2F_6 \rightarrow CF_3 + CF_3 + e$	2.10(− 13)	b,15,16
$e + C_2F_4 \rightarrow CF_2 + CF_2 + e$	2.10(− 13)	b,15,16
$e + CF_3 \rightarrow CF_2 + F + e$	2.10(− 13)	b,15,16
$e + CF_2 \rightarrow CF + F + e$	2.10(− 13)	b,15,16
$CF_2 + h\nu \rightarrow CF_2$	2.00(− 17)cm^2	9
$CF_3^- + M^+ \rightarrow CF_3 + M$	1.00(− 06)	c
$F^- + C_nF_m^+ \rightarrow F + C_nF_m$	1.00(− 06)	c
$Kr^+ + CF_4 \rightarrow CF_3^+ + F + Kr$	1.00(− 11)	19
$Kr_2^+ + CF_4 \rightarrow CF_3^+ + F + Kr + Kr$	1.00(− 11)	d,19
$Ar^+ + CF_4 \rightarrow CF_3^+ + F + Ar$	9.58(− 10)	e,20
$Ar_2^+ + CF_4 \rightarrow CF_3^+ + F + Ar + Ar$	9.58(− 10)	d,20
$CF^+ + CF_4 \rightarrow CF_3^+ + CF_2$	3.30(− 10)	19
$CF^+ + C_2F_6 \rightarrow C_2F_5^+ + CF_2$	3.90(− 11)	19
$CF^+ + C_3F_8 \rightarrow C_3F_7^+ + CF_2$	7.10(− 11)	19
$CF_3 + F \rightarrow CF_4$	2.90(− 11)	f,15,26
$CF_2 + F \rightarrow CF_3$	1.30(− 11)	f,15
$CF + F \rightarrow CF_2$	1.00(− 11)	f,15
$C_2F_4 + F \rightarrow CF_3 + CF_2$	4.00(− 11)	15
$C_2F_5 + F \rightarrow CF_3 + CF_3$	1.00(− 11)	15
$CF + CF_2 \rightarrow C_2F_3$	1.00(− 12)	f,15
$C_2F_3 + F \rightarrow C_2F_4$	1.00(− 12)	f,15
$CF_2 + CF_3 \rightarrow C_2F_5$	1.00(− 12)	f,15
$CF_3 + F_2 \rightarrow CF_4 + F$	1.04(− 12)	25
$CF_2 + CF_2 \rightarrow C_2F_4$	5.00(− 14)	f,15,28
$CF_3 + CF_3 \rightarrow C_2F_6$	8.30(− 12)	3,15
$CF_3^- + F \rightarrow CF_3 + F^-$	5.00(− 08)	29
$Ar^* + CF_4 \rightarrow CF_2 + F_2 + Ar$	4.00(− 11)	g,5
$Ar^{**} + CF_4 \rightarrow CF_2 + F_2 + Ar$	4.00(− 11)	d,5
$Ar^* + C_2F_6 \rightarrow CF_3 + CF_3 + Ar$	4.00(− 11)	d,5
$Ar^{**} + C_2F_6 \rightarrow CF_3 + CF_3 + Ar$	4.00(− 11)	d,5
$Ar_2^* + CF_4 \rightarrow CF_2 + F_2 + Ar + Ar$	4.00(− 11)	d,5
$Ar_2^* + C_2F_6 \rightarrow CF_3 + CF_3 + Ar + Ar$	4.00(− 11)	d,5
$Kr^* + CF_4 \rightarrow CF_4 + Kr$	7.00(− 13)	5
$Kr^{**} + CF_4 \rightarrow CF_4 + Kr$	7.00(− 13)	d,5
$Kr^* + C_2F_6 \rightarrow C_2F_6 + Kr$	7.00(− 13)	d,5
$Kr^{**} + C_2F_6 \rightarrow C_2F_6 + Kr$	7.00(− 13)	d,5
$Kr_2^* + CF_4 \rightarrow CF_4 + Kr + Kr$	7.00(− 13)	d,5
$Kr_2^* + C_2F_6 \rightarrow C_2F_6 + Kr + Kr$	7.00(− 13)	d,5
$KrF(B) + C_nF_m \rightarrow Kr + F + C_nF_m$	1.00(− 11)	h
$ArF(B) + C_nF_m \rightarrow Ar + F + C_nF_m$	2.00(− 11)	h

[a]Units of cm^3 s^{-1} unless noted.
[b]Rate coefficient was obtained from the Monte Carlo simulation for slowing of an e-beam in an Ar/Kr/F$_2$/CF$_4$ mixture using the CF$_4$ cross sections in Ref. 16, and branching ratios in the cited reference.
[c]Estimated. See Ref. 12 for analogous reactions.
[d]Estimated. See text and cited reference.
[e]Value is the maximum in the range of uncertainty.
[f]Rate constant for a three-body association reaction is quoted using its equivalent two-body rate coefficient at 1.75 atm. See Ref. 15.
[g]See text for discussion of branching.
[h]Estimated. See text for discussion.

W values used in our model were obtained from a separate Monte Carlo simulation for the slowing of e beams in high-pressure gases.[14]

In addition to the conventional plasma chemistry of e-beam-pumped KrF lasers, we included reactions pertaining to the plasma decomposition of CF$_4$. These reactions are listed in Table I. The choice of reaction pathways was largely based on the work of Plumb and Ryan who have investigated the plasma kinetics of CF$_4$ in the context of the etching of silicon.[15] Rate coefficients for electron-impact processes for CF$_4$ were also obtained from the Monte Carlo simulation by including the cross sections derived by Hayashi.[16]

The threshold for electron-impact dissociation of CF$_4$ is $\simeq 12.5$ eV.[17] Therefore quenching reactions of CF$_4$ with Ar (4s) and Ar* are capable of dissociating the fluorocarbon whereas those with Kr (5s) and Kr* are not likely to dissociate the molecule. As a worst case, we therefore assumed that quenching reactions of CF$_4$ with Ar* and Ar$_2^*$ are dissociative resulting in CF$_2$ + 2F. We included no branching to KrF(B) or ArF(B) for quenching reactions of Kr*, Kr$_2^*$, Ar*, or Ar$_2^*$ with C$_n$F$_m$ species, in analogy with measurements for quenching of Kr (5s) by CF$_4$ made by Velazco and co-workers.[5] Branchings to KrF(B) and ArF(B) have been measured for ion neutralization between Kr$^+$ and Ar$^+$ with SF$_6^-$, showing that this type of process can result in excitation of the upper laser level when complex ions are reactants.[18] As a worst case, though, we assumed that neutralization of Kr$^+$ and Kr$_2^+$ with CF$_3^-$ is nondissociative with no branching to KrF(B).

The thermochemistry of CF$_4$ and CF$_3^+$ implies that charge exchange reactions with Ar$^+$ are exothermic by 1.1 eV whereas those with Kr$^+$ are endothermic by 0.76 eV.[20] Charge exchange reactions of Ar$^+$ with CF$_4$ producing CF$_3^+$ have been measured at thermal energies yielding a gas kinetic rate coefficient [7(±2.6) $\times 10^{-10}$ cm^3 s^{-1}].[20] A smaller rate coefficient has been measured for charge exchange with Kr$^+$ (1×10^{-11} cm^3 s^{-1}).[19] In analogy to these results, we assumed that all charge-exchange reactions between Kr$^+$, Kr$_2^+$, Ar$^+$, and Ar$_2^+$ have a unity branching to CF$_3^+$ with rates given by the monomer species.

Rate coefficients for the quenching of KrF(B) and ArF(B) by CF$_4$ are not presently available. The coefficients for quenching of XeF(B) and XeCl(B) by CF$_4$ are 0.5×10^{-12} (Ref. 21) and 2.5×10^{-12} cm^3 s^{-1},[22] respectively. These values are small due to the absence of acceptor states in CF$_4$ at the energies of XeF(B) (3.5 eV) and XeCl(B) (4.0 eV). One normally expects, though, that the rate coefficients for quenching under these conditions will increase with increasing energy of the rare-gas halide, as shown by the increase in the rate coefficient of XeCl(B) compared to XeF(B). We therefore estimated as a worst case that the rate coefficients for quenching of KrF(B) (energy of 5 eV) and ArF(B) (energy at 6.5 eV) by CF$_4$ are 1.0×10^{-11} and 2.0×10^{-11} cm^3 s^{-1}, respectively. As discussed below, even with these values quenching of the excimer species by CF$_4$ has a small effect on the performance of the laser.

III. RESULTS FOR CONTAMINATION OF KrF LASERS BY CF4

The pumping conditions we chose were Ar/Kr/F$_2$ = 95/5/0.5 at 1.75 atm with a power deposition of 100–400 kW cm^{-3} having a current pulse of 400 ns (FWHM).
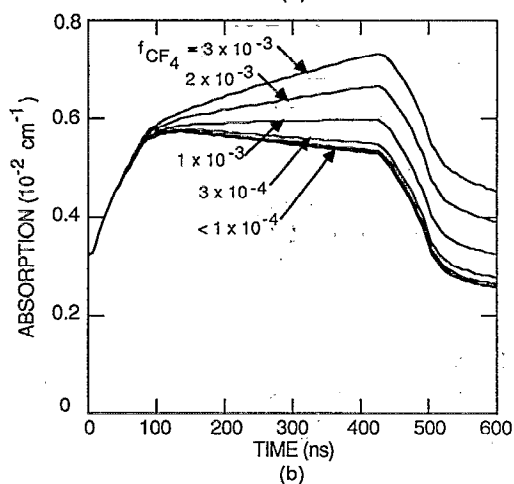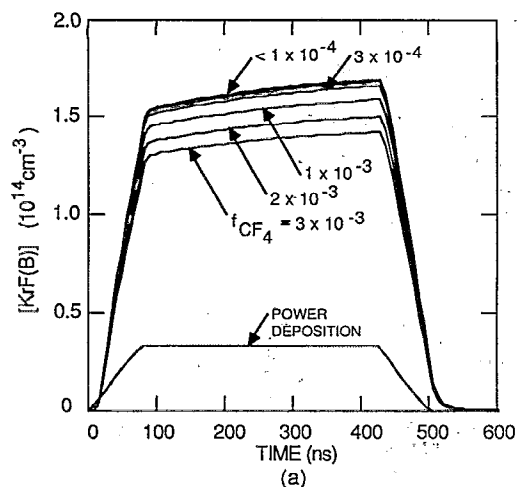
FIG. 1. Laser parameters for e-beam excitation of a 1.75-atm Ar/Kr/$F_2 = 95/5/0.5$ gas mixture for different levels of CF_4 impurity: (a) KrF(B) density and (b) total absorption. The power deposition is 250 kW cm^{-3}. f_{CF_4} is the mole fraction of CF_4 in the mixture. Absorption increases during the current pulse due to buildup of CF_2.

FIG. 2. Laser parameters for the conditions of Fig. 1: (a) CF_2 density and (b) g_0/α (small signal gain/absorption). The increase in CF_2 during the pulse causes a decrease in g_0/α. This quantity usually increases due to burnup of F_2.

These values are similar to those used in the larger aperture module (LAM) laser system at Los Alamos National Laboratory whose performance history motivated this work.[23] The computed density of KrF(B) and absorption α as a function of time are shown in Fig. 1 for a power deposition of 250 kW cm^{-3} and various levels of CF_4 contamination. The density of KrF(B) we obtained in a pristine mixture corresponds to a small signal gain of ≈ 0.04 cm^{-1} for a stimulated emission cross section of 2.6×10^{-16} cm^2.[12] This gain, and absorption of 0.055 cm^{-1}, agree well with recent and past measurements of these quantities in e-beam-excited KrF lasers. These experimental results are summarized in Ref. 24.

For a power deposition of 250 kW cm^{-3} and pulse length of 400 ns (≈ 100 J/1) the CF_4 is not significantly depleted. The KrF(B) density increases moderately during the current pulse as F_2, a dominant quencher, is depleted by dissociative attachment reactions. With CF_4 contamination, the density of KrF(B) is uniformly decreased during the current pulse, with a significant decrease occurring when the mole fraction of CF_4, $f(CF_4)$, exceeds 3×10^{-4},
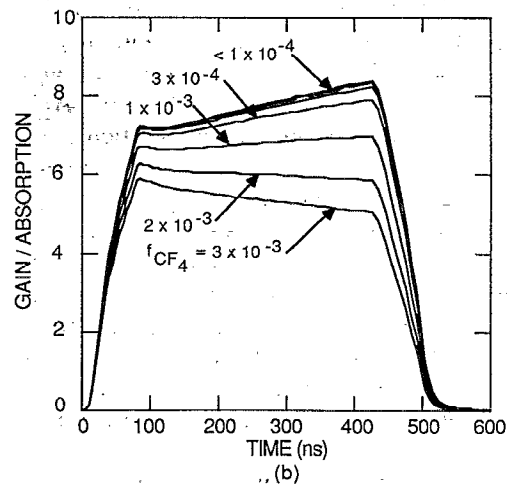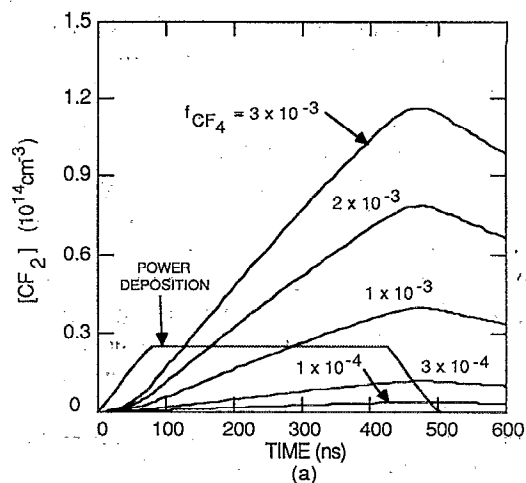
or 300 ppm. This trend indicates that CF_4 is the dominant quenching or intercepting species which reduces KrF(B) as opposed to its dissociation products. F_2 accounts for approximately 60% of the total absorption at 248 nm in the absence of contamination. The absorption normally decreases during the current pulse due to burnup of F_2 (approximately 25% in these cases). In the presence of contamination, absorption increases both as the CF_4 concentration increases and as the current pulse progresses. These results imply that products of CF_4 that are relatively unreactive and accumulate during the current pulse contribute to the incremental increase in absorption. The contamination threshold for CF_4 to produce significant additional absorption is greater than hundreds of ppm.

The species responsible for the increase in absorption in our reaction scheme is CF_2, whose density is shown in Fig. 2(a) for the same conditions as in Fig. 1. Densities in excess of 10^{13} cm^{-3} are generated in heavily contaminated systems [$f(CF_4) > 10^{-4}$]. Although CF_3^- is absorbing at 248 nm, the rate constant for dissociative attachment to CF_4 is small ($\approx 5 \times 10^{-12}$ cm^3 s^{-1}) due to the high

threshold energy for the process (≈ 5 eV).[6] CF_3^- also charge exchanges with F. The density of CF_3^-, therefore, does not exceed 10^{10} cm^{-3} for most conditions and consequently does not contribute significantly to absorption.

CF_2 is relatively unreactive in this mixture since it does not react exothermically with CF_4. Therefore, the density of CF_2 tends to integrate during the current pulse. As a result of CF_2 accumulating during the current pulse the absorption also increases, resulting in a decrease in g_0/α (small signal gain/absorption), as shown in Fig. 2(b). Since g_0/α is a measure of the extraction efficiency, the laser efficiency also decreases during the current pulse (see below). The dominant sink for CF_2 is association reactions with atomic fluorine to form CF_3 ($CF_2 + F \rightarrow CF_3$), having approximately a 300 ns time constant. This reaction can be followed by a second reassociation reaction to reform CF_4 ($CF_3 + F \rightarrow CF_4$). CF_2 therefore recycles to some degree during the pulse, being reformed by electron impact and dissociative excitation transfer to CF_3 and CF_4 from Ar*. Although CF_2 and CF_3 undergo mutual association reactions to form C_2F_4, C_2F_5, and C_2F_6, the rate constants for these processes are not particularly large ($< 10^{-12}$ cm^3 s^{-1}). Since the density of F ($10^{16}-10^{17}$ cm^{-3}) greatly exceeds that of CF_2 and CF_3 ($< 10^{14}$ cm^{-3}) association reactions with F are most important in determining the density of CF_2. C_2F_5 and C_2F_4 also back react with F atoms with moderate rate coefficients ($> 10^{-11}$ cm^3 s^{-1}) to form CF_2 and CF_3. The end result is that C_2F_6 is the only fluorocarbon dimer having a density that exceeds 10^{13} cm^{-3} when $f(CF_4)$ is larger than 10^{-3}. C_2F_6 is not chemically reactive (other than being dissociated by electron impact or excitation transfer) and is not absorbing at 248 nm.[8] Therefore, it is not additionally harmful.

Laser power as a function of time and laser power efficiency at 425 ns as a function of $f(CF_4)$ are shown in Fig. 3 for the same conditions discussed above. As the level of CF_4 contamination increases, the laser power switches from increasing during the current pulse to decreasing during the current pulse. This trend is a consequence of the accumulation of CF_2 which reduces g_0/α during the current pulse. This compensates the burnup of F_2 which otherwise results in g_0/α increasing during the current pulse. Laser power efficiency η consequently decreases from 6.4% in a pristine mixture to 3.8% for $f(CF_4) = 3 \times 10^{-3}$. This yields an incremental slope efficiency of $\beta = (\Delta\eta/\eta)/\Delta f(CF_4)$ of -2×10^{-4}/ppm. Kimura and Seamans obtained a similar sensitivity of $\beta = -3 \times 10^{-4}$ in their measurements of laser efficiency in an e-beam-pumped XeF laser during controlled addition of impurities.[4]

To identify the kinetics dominantly contributing to the reduction in KrF(B) density, we defined two processes: direct interception and indirect interception. Indirect interception processes are those in which CF_4 quenches a high-order precursor to forming KrF(B). For example,

$$ArF(B) + CF_4 \rightarrow Ar + F + CF_4 \tag{1}$$
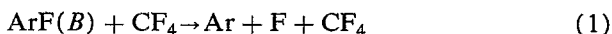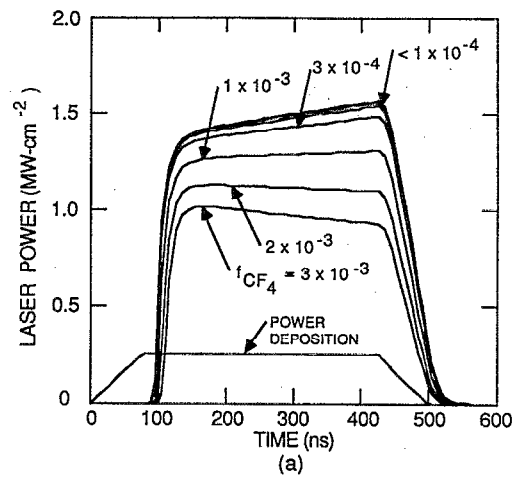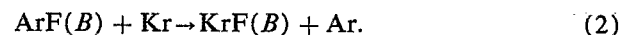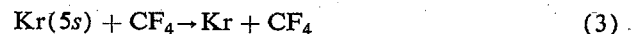
is an indirect interception reaction of the process
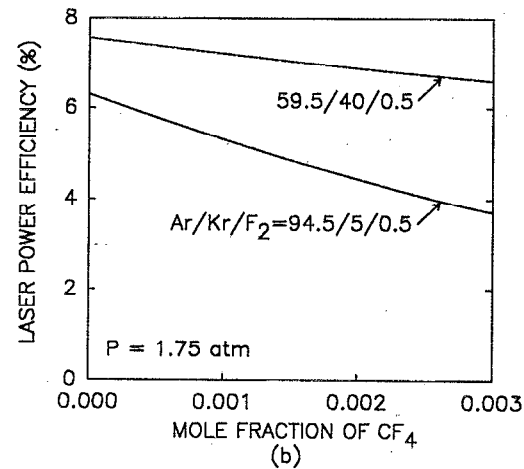
FIG. 3. Laser performance for different levels of CF_4 contamination: (a) laser power and (b) laser power efficiency at 425 ns. The conditions are nominally the same as for Fig. 1. Laser power efficiency is shown for two gas mixtures, showing that mixtures having higher proportions of Kr are less sensitive to CF_4 contamination.

$$ArF(B) + Kr \rightarrow KrF(B) + Ar. \tag{2}$$

A direct interception reaction quenches a first-order reactant leading to KrF(B). For example the quenching process

$$Kr(5s) + CF_4 \rightarrow Kr + CF_4 \tag{3}$$

would intercept directly the reaction

$$Kr(5s) + F_2 \rightarrow KrF(B) + F. \tag{4}$$
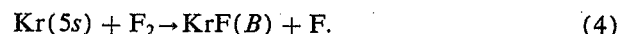
Indirect interception most often involves quenching of Ar-excited species whereas direct interception involves quenching of Kr excited species. Direct quenching reactions are collisions of CF_4 and its reaction products with KrF(B).

The density of KrF(B) is plotted in Fig. 4 as a function of time during the current pulse with and without CF_4 as an impurity [$f(CF_4) = 2.0 \times 10^{-3}$]. Results are also shown for excluding indirect interception reactions, direct interception reactions, and direct quenching reactions, respectively. Direct quenching of KrF(B) by CF_4 contrib-
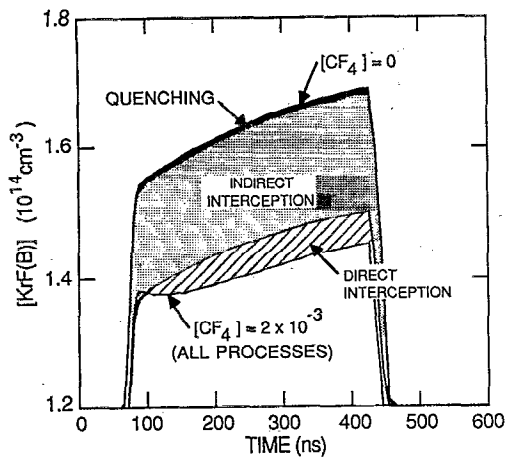
FIG. 4. KrF(B) density during the current pulse with and without contamination by CF_4 (mole fraction of 2×10^{-3}). Results are shown for excluding direct interception, indirect interception, and quenching reactions. The cross-hatched and dotted regions shown the contributions of quenching and indirect interception to the decrease in the KrF(B) density.
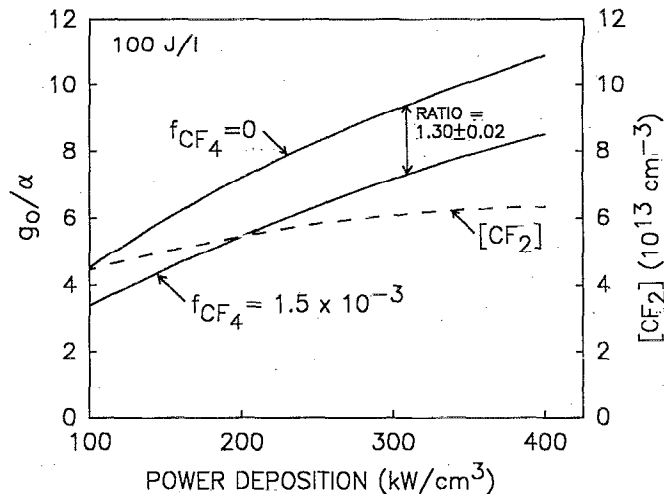
FIG. 5. g_0/α (small signal gain/absorption) and CF_2 density for various power depositions having a total energy deposition of 100 J/l. Results are shown with and without CF_4 contamination The ratio between these values is nearly constant, showing that energy deposition is the scaling parameter of interest for contamination by CF_4.

utes only a few percent to the decrease in the density of the upper laser level. Direct interception of Kr*, Kr^+, Kr_2^+, and F^- causes a larger, but moderate decrease. The majority of the decrease in the density of KrF(B) results from indirect interception reactions. This trend results from the rates of quenching of Ar* being larger than those for Kr*, and because the density of Ar precursors are more numerous. Gas mixtures having large fractions of Kr should therefore be less sensitive to contamination by CF_4. This result is shown in Fig. 3(b) where laser power efficiency is plotted as a function of $f(CF_4)$ for an Ar/Kr/F_2 = 59.5/40/0.5 mixture. The decrease in laser efficiency is less than a third that in the leaner mixture.

A large portion of the deleterious effects of contamination by CF_4 results from accumulation of CF_2. For a given pumping rate absorption increases as the pulse length, and energy deposition, increase. When the power deposition is increased the rate of generation of CF_2 increases but so does the gain. There is, then, a compensating effect on g_0/α. The relative amount that g_0/α decreases should not change significantly as power deposition is varied. The scaling parameter of interest should then be energy deposition because for short pulses ($< 1\ \mu$s) the density of CF_2 is ultimately determined by energy deposition and not power deposition. This scaling is shown in Fig. 5 where g_0/α and [CF_2] are each plotted as a function of power deposition for a fixed energy deposition of 100 J/l. Values are shown for a pristine mixture and with $f(CF_4)$ = 1.5×10^{-3}. The density of CF_2 does not change significantly at a fixed energy deposition for power depositions of 100–400 kW cm^{-3}. g_0/α increases with increasing power deposition, but the ratio between the pristine and contaminated mixtures remains nearly the same.

IV. CONCLUDING REMARKS

The performance of e-beam-excited KrF lasers contaminated by CF_4 has been theoretically investigated. We find that impurity levels in excess of hundreds of ppm are required to significantly degrade laser efficiency. KrF lasers are similar to XeF lasers in this respect.[4] The density of KrF(B) decreases with increasing CF_4 density primarily due to quenching of excited states of argon, which are indirect precursors to formation of the upper laser level. Absorption increases due primarily to the accumulation of CF_2 during the current pulse. The cited impurity levels are large compared to the values believed to be present in e-beam-pumped laser systems showing symptoms of fluorocarbon contamination,[30] though they are commensurate with that which might be found in some discharge-excited systems. This leads one to suspect that other trace fluorocarbon impurities have either a greater quenching or intercepting capability, or generate CF_2 by plasma decomposition at a higher rate than CF_4.

Due to the high threshold energy for dissociation of CF_4 (12.5 eV) and the low electron temperature in e-beam-pumped lasers ($T_e \simeq 1$ eV), the rate of electron impact dissociation of CF_4 by bulk electrons is low. Electric discharge lasers, however, have electron temperatures of 4–5 eV. The rate of dissociation of CF_4, as well as other fluorocarbon impurities, will be many times larger in discharge-excited lasers than that in e-beam-pumped systems. One can, therefore, expect electric discharge KrF lasers to be more sensitive to CF_4 than e-beam-pumped lasers having the same degree of contamination.

ACKNOWLEDGMENTS

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