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Internal structuring of gallium arsenide using short laser pulses

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Abstract: Laser writing inside semiconductors attracts attention as a possible route for three-dimensional integration in advanced micro technologies. In this context, gallium arsenide (GaAs) is a material for which the best conditions for laser internal modification (LIM) have not been established yet. We address this question by using laser pulses at a fixed wavelength of 1550-nm. A large parameter space is investigated including the response to the applied pulse energy, pulse duration (from femtosecond to nanosecond) and the focusing conditions. We report that well-defined and reproducible internal modifications are achievable with tightly focused nanosecond pulses. The measured writing thresholds are systematically compared to those obtained in silicon (Si), a more extensively studied material. In comparison to Si, we also observe that GaAs is more prone to filamentation effects affecting the modification responses. The reported specific observations for LIM of GaAs should facilitate the future process developments for applications in electronics or photonics.

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1. Introduction

Laser internal modification (LIM) of transparent materials has been widely investigated because of its unique capability to directly create three-dimensional devices without affecting their surfaces. In the past decades, LIM has been successfully applied in dielectric materials [1], generating tangible impact in various domains including integrated photonics [2], lab-on-a-chip [3], optical fiber micro-sensors [4], and data storage [5]. Recently, the target materials have been extended from wide-bandgap dielectrics to more challenging narrow bandgap semiconductor materials, including in particular the most important semiconductor: silicon (Si) [6]. In comparison to dielectrics, researches [7–12] have revealed extremely severe requirements on the applied laser conditions to obtain reproducible and highly localized modification with ultrashort pulses inside Si. Among the identified specificities that degrades the achievable energy densities at focus inside narrow gap materials, one can mention their high refractive index causing a strong refraction at air-material interface thus reducing the achievable focusing angle. More importantly, their generally high multiphoton ionization coefficients and nonlinear refractive index usually causes severe beam distortions by strong nonlinear propagation [13] and plasma effects far prior to focus [14]. To overcome these obstacles, various solutions have been proposed very recently to improve the energy density at focus and successfully achieve internal modifications [7,8,15–18]

While efforts have concentrated on investigating LIM of Si in the last few years, there are very little work on other important semiconductors, including gallium arsenide (GaAs) which is widely used in areas such as light-emitting diodes, solar cells, integrated circuits, and wavelength conversion. LIM of GaAs offer new process capabilities for these applications, including welding [19], dicing [20] or refractive index engineering by the same way that it is today exploited in glass [1–4]. However, up to now, there are very few works investing LIM of GaAs [15,21]. M. Mori et al. [15] have observed internal modification in GaAs by using double-pulse approach

using a femtosecond laser at 1.24 μm wavelength. This work reports a threshold condition in this specific case with a comparison to Si but not to other irradiation conditions (e.g., longer pulse-duration regimes). Later O. Tokel et al. [21] have observed the formation of nanograting inside the material. However, the study was focused on the nanosecond regime leaving a large gap with the previous work using ultrafast lasers [15]. In principle, it cannot be directly expected that LIM of GaAs is easier than Si according to the material properties. A brief comparison of relevant properties of GaAs and Si is shown in Table 1. Some similar properties intrinsic to semiconductors are shown, such as narrow bandgaps and high refractive indices. Meanwhile, GaAs has a direct bandgap that can lead to easier absorption of photons by avoiding mismatch of crystal momentum like in Si which exhibits an indirect bandgap. Correspondingly, its two-photon absorption coefficient is around 10 times higher than that of Si. Accordingly, a lower laser intensity is expected to deposit the same amount of energy in GaAs as compared to Si. However, GaAs has three times larger nonlinear refractive index than Si. Therefore, larger Kerr-induced effects are also expected for GaAs at reduced laser power. With these and other important differences of material properties and the current knowledge on LIM of semiconductors, one can intuitively expect strong changes in the complex interplay between the nonlinear processes, thus the modification response can differ when translating the situation from Si to GaAs.

Table 1. Comparison of relevant properties of GaAs and Si (at 1.55 μm)

Basic Optical Parameters	GaAs	Si	Reference No.
Bandgap (in eV at 300 K)	1.43	1.12	
Bandgap type	Direct	Indirect	
Two Photon Absorption coefficient (in cm/GW)	7	0.7	[22–24]
Refractive index	3.38	3.48	
Nonlinear refractive index ^a (in cm^2/GW)	1.5×10^{-4}	5.6×10^{-5}	[23,25]

^aIt is worth noting that these data are typical values that may differ depending on the measurement conditions.

In this paper, we systematically investigate LIM of GaAs at fixed wavelength by using laser pulses with a wide range of durations from the femtosecond to nanosecond regime. In this way, we identify the required conditions that can be exploited for successful writing applications. These must serve as important guidelines for future LIM-based 3D fabrication methods for GaAs applications. The results obtained in this large parametric space on GaAs are systematically compared to the LIM results of Si using the same experimental conditions [10]. Such comparative study sheds light on the important material properties influencing LIM results.

2. Experimental setup

The overall experimental setup is shown in Fig. 1. Several sources are used to carry out investigations with different pulse durations, but they all operate at the same central wavelength of 1550 nm to permit direct comparisons without considering any spectral dependency. First, ultrashort laser pulses with a duration < 190 fs are produced by an Ytterbium source (Pharos, Light-conversion) before injection in an optical parametric amplifier (Orpheus, Light-conversion) for conversion at wavelength of 1550 nm [10]. While GaAs crystal is in principle transparent to 1030 nm, the conversion to 1550 nm permits avoiding residual absorption by potential deep-level impurities typically introduced into GaAs crystal during its growth. This choice also facilitates the comparisons to previous studies performed in the domain of full transparency for silicon. In addition, it also avoids complications associated change of laser sources and/or optical elements if different wavelengths are used. Then, the energy of the beam is controlled by the combination of a half-wave plate (HWP) and a polarizer (PLZ). The beam energy can be directed to two

possible paths. In one path the beam is dispersed using a parallel grating pair arrangement (not shown, ‘Pulse stretcher’ box in Fig. 1, the details can be found in Ref. [10]) in order to produce picosecond pulses ranging from 4 to 21 ps depending to the grating separation distance (pre-calibrated by autocorrelation measurements). As can be seen in Fig. 1, an alternative path after the beam splitter (BS) directs the femtosecond laser beam directly to the focusing objective (OB1). To investigate the material response in the nanosecond regime, a nanosecond fiber laser source (MWTech, PFL1550) with ~ 5 ns pulse duration is added. According to the specifications from the laser suppliers, the M2 is between 1.1 and 1.5 for the nanosecond beam and less than 1.3 at the Orpheus output (femtosecond and picosecond beams). These beam qualities were confirmed by the measured focusing power on air comparing well with theoretical predictions. However, we did not find any potential beam imperfections influencing the propagation features reported in this work. The nanosecond laser beam from the laser source has a relatively large divergence, so they are injected into a telescope consisting of a lens pair ($f = 100$ mm and $f = 200$ mm, not shown in the figure) to adjust the beam size and minimize the divergence. Gold mirrors (M) and BS have been used so that all beams are prepared with similar sizes (diameter around 5.2 mm) and can be used for writing investigations with the same focusing objective. The focusing characteristics are also measured in air by a high-resolution imaging system (not shown in Fig. 1) to ensure that all the beams can be focused with near-identical spot sizes at the same position. For irradiation with individual pulses of different pulse durations, beam dumps (BD) are introduced in each path for beam selectivity.

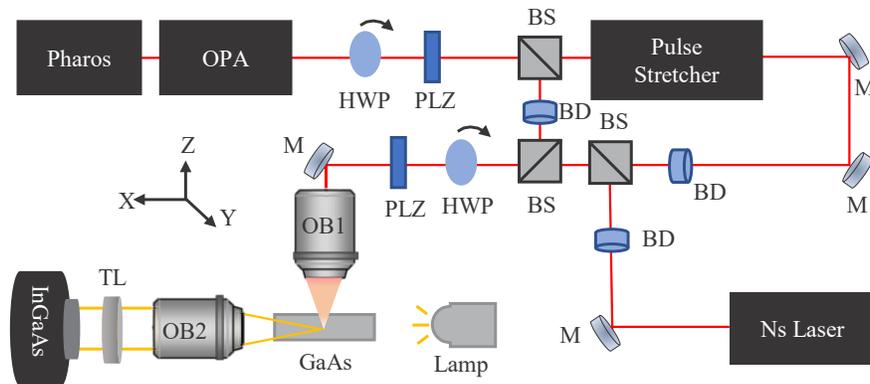


Fig. 1. Schematic of experimental setup for studying LIM inside GaAs. Abbreviation: OPA for optical parameter amplifier; HWP for half-wave plate; PLZ for polarizer; M for mirror; BD for beam damper (removable according to desired beam on target); BS for beam splitter; OB for objective; TL for tube lens; InGaAs for InGaAs camera.

For the irradiation experiments, we use several infrared objective lenses (OB1, Olympus LMPLN-IR/LCPLN-IR) of $NA = 0.3(10\times)$, $0.45(20\times)$, $0.65(50\times)$ and $0.85(100\times)$. The lenses with $NA > 0.3$ are equipped of correction collars to compensate the spherical aberration induced by the refractive index mismatch at air/Si interface. The value of the corrected collar is systematically adjusted based on the applied focusing depth in the samples. As the refractive index of GaAs is nearly identical to Si, we use the same corrections as if we were working in Si. For getting maximum focusing power, the beams overfill the entrance pupil of the objective lenses ($NA = 0.45$, 0.65 and 0.85) at the exception of the 0.3 NA tested case. The spot sizes obtain with each lens is given in Ref. [10].

GaAs wafers (Neyco, VGF growth method, intrinsic) of $\langle 100 \rangle$ crystal orientation with a thickness of $600 \mu\text{m}$ are cleaved to prepare square samples (about 1 cm). The wafers are polished and cleaned to guarantee optically flat surfaces and to avoid any surface effect which could

influence the laser conditions inside the samples. The laser pulses are focused 300 μm under the surface. For systematic comparisons, the modifications are obtained by repeatedly irradiating the samples with 1000 pulses obtained by 1 second exposure time at repetition rate of 1 kHz (controlled by a pulse picker). Given the toxicity of GaAs, appropriate safety measures are required for the manipulation and laser processing of the samples. With the internal nature of the processing zones in this study, we limited these measures to the wearing protective clothing (incl. gloves). However, careful attention is paid to avoid surface ablation when positioning the beam focus inside the materials.

For detecting and observing the bulk modifications inside GaAs, an in-situ infrared microscopy system is employed. It is based on a custom arrangement with a long working distance objective lens (OBJ2, Mitutoyo 20 \times), a tube lens (TTL200MP, Thorlabs), an InGaAs IR camera (Raptor, OWLSWIR 640), and a tungsten-halogen lamp (Thorlabs, QTH10). The spatial resolution of each acquired images is theoretically estimated at 0.75 μm . Accordingly, to this limit, we do not expect to detect the first material changes but contrasted modifications reaching micrometer size can be readily observed. This is an important consideration for the threshold determinations.

3. Results and discussions

3.1. Pulse-duration dependence of LIM

First of all, we compare the conditions for LIM of GaAs using laser pulses of different pulse durations. The irradiations are conducted by using the same microscope objective of NA = 0.85. This corresponds to the strongest focusing conditions and so it offers the best possibilities to exceed the modification threshold in comparison to lower NA conditions. Using the first beam path (see above), the sub-190-fs laser pulses are directly applied for tentative modification studies inside GaAs sample. However, we have observed that no internal structure could be produced with this pulse duration up to the maximum tested energy of 90 μJ . This observation is very similar to the one of our previous reports investigating similar configurations for LIM of Si [10]. Previous works in Si indicate that longer pulses are required to deliver energy densities above modification threshold inside Si [10]. Therefore, we have tested the modification of GaAs by using pulses of 21 ps and ~ 5 ns. The results are compared in Fig. 2(a) where infrared lateral microscopy is used to directly observe the changes in modification morphologies depending on the applied conditions. For each tested pulse energy, the irradiations are also repeated 10 times for damage probability analyses near threshold conditions. First, we note that internal modification is achievable using these laser conditions (see top images). However, obvious differences can be observed between the modification results obtained with the two pulse durations.

An important observation is the difference of the modification sizes. The modifications induced by picosecond laser pulses tend to exhibit a longer length (along the laser propagation direction, longitudinal writing resolution) as directly revealed with Fig. 2(a). At the pulse energy of 30 nJ, the lowest tested energy leading to unity damage probability in the 21-ps case, the average length of the modifications is 41.6 μm with a standard deviation of 16.2 μm . This length is much longer than the confocal parameter of the focus, which is around 13.2 μm considering a linear propagation case (simulated by using vectorial theory [26]). For nanosecond pulses as shown in Fig. 2(b), the modifications above threshold are clearly much shorter and more stable both in terms of spatial dimensions and amplitude of changes. At the lowest tested energy (13.2 nJ) with unity damage probability, the average length is 15 μm with a standard deviation of 0.8 μm . This length is comparable to the theoretical Rayleigh range for the applied beam.

Another important observation is on the repeatability of the modifications at different pulse energies. The ratios between the occurrence of modifications and attempts are shown in the damage probability curves represented in Fig. 2(b). For picosecond laser pulses, one notes a very stochastic response and non-reproducible modifications. The transition from 0% to 100% damage probability is spanning over approximately two orders of magnitude (from 0.3 nJ to 30 nJ). This

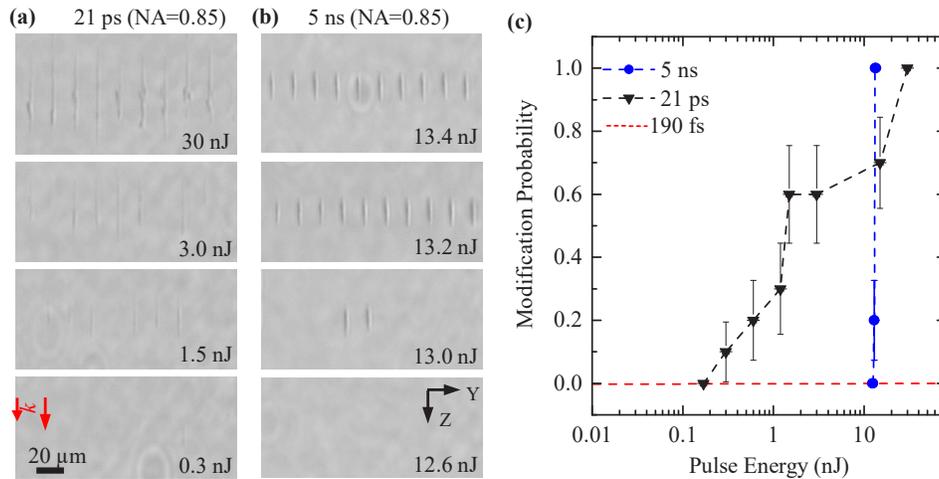


Fig. 2. (a),(b) Bulk modifications in GaAs obtained under NA = 0.85 focusing conditions with pulses of 21-ps (a) and ~5-ns pulse duration (b). The laser propagation direction is given by the wavevector (\vec{k}). (c) Modification probability graph for bulk modifications in GaAs at the tested pulse durations. Statistical errors (along Y-axis) are calculated by the following relation $\sqrt{P \cdot (1 - P) / N}$ where P is the probability and N is size of statistical samples (number of irradiated sites).

large energy-range of instability cannot be explained by the fluctuations of the laser system used in the experiment. The measured pulse-to-pulse energy fluctuation measured to be less than 3% in this case. As a comparison, the 0-100% transition with the nanosecond pulses is extremely abrupt according to a damage probability at 0% measured with pulse energy of 12.6 nJ while 100% is already observed at pulse energy of 13.4 nJ. This leads to the uncommon observation of a more deterministic response for longer pulses, as it is the contrary to the widely studied response of dielectrics [27,28]. While the underlying physical aspects behind this observation remains partially understood (see after), this reveals another important specificity of semiconductors (in comparison to dielectrics) as it was also observed for silicon [10,29].

These differences show that the pulse duration is an important parameter for obtaining reproducible and controlled modifications. The pulse duration leads to a drastic change of peak power, and a peak power dependent phenomenon is the self-focusing effect, which is expected to play an important role for the material modification results. An estimate of the critical power for self-focusing of a collimated Gaussian beam is given by [30]: $P_{cr} = \lambda^2 / (2\pi n_0 n_2)$, where n_0 and n_2 are the linear and nonlinear part of the refractive index. For a wavelength of 1550 nm, taking $n_0 = 3.38$ and $n_2 = 1.5 \times 10^{-4} \text{ cm}^2/\text{GW}$ (see Table 1) leads to a critical power of only 7.6 kW in GaAs. This is corresponding to a pulse energy of 230 nJ for 21-ps pulses and 54 μJ for 5-ns pulses after considering the Fresnel losses at the interface. While the picosecond pulse energies found at 100% probability are approaching this estimated value, all tested nanosecond pulse energy conditions remain several orders of magnitude below this critical value. Accordingly, one can safely exclude a significant self-focusing contribution on potential propagation perturbations for the nanosecond irradiations. Oppositely, the observed features in the picosecond regime potentially originate from filamentary effects caused by a balance between significant self-focusing and plasma defocusing contributions [31]. The resulting formation of long filaments of deposited energy densities, subject to this balance between nonlinear processes, makes then the process more prone to laser instabilities and irregularities in the experimental conditions (incl. materials). This explains the high energy-range of non-reproducible modifications and irregular shape of the

obtained modifications. Some similar observations made with ultrashort pulses in transparent materials are also observed under specific conditions [31].

3.2. NA dependence of LIM

The results in previous section show that nanosecond laser pulses give more repeatable modifications than shorter pulses. In this section, we conduct similar modification experiments by using looser focusing conditions from NA = 0.65 to NA = 0.3.

First, we have tested the modification response with 21-ps pulses using looser focusing conditions. We find no bulk damage can be achieved at lower NA conditions from 0.65 to 0.3. This is again similar to the results obtained when studying LIM of Si and showing the requirement of a minimum NA of 0.85 for successful modifications [10]. Previous works on Si reveals the strong NA requirements on the focusing conditions for circumventing prior-to-focus detrimental interactions and achieving enough laser energy density near focus for internal structuring with ultrashort pulses of high temporal contrast [9,32]. By finding here very similar requirements in GaAs, this work confirms that it is likely a common feature for most narrow-gap semiconductors exhibiting strong propagation nonlinearities.

Then, we have tested the NA dependence of modifications produced with the nanosecond pulses. The results are shown in Fig. 3(a). Modifications are achievable with NA = 0.65 and NA = 0.45 whereas no damage has been detected with NA = 0.3, an aspect that can be reasonably attributed to the limitation of the maximum pulse energy of 5 μJ on target with the compact nanosecond laser source used in this work. Figure 3(a) shows the modification results of NA = 0.65 and NA = 0.45. Compared to NA = 0.85 (see Fig. 2), the modifications require higher energies to exceed the damage threshold. This is commonly expected because lower NA leads to looser focusing and reduced delivered energy densities.

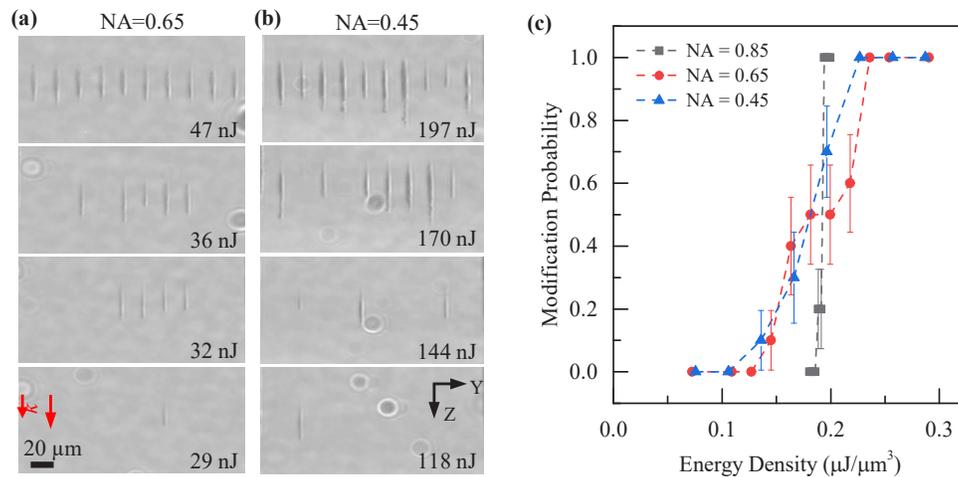


Fig. 3. Repeatability tests of bulk modifications at a depth of 300 μm inside GaAs using 5 ns pulses focused with (a) NA = 0.65 and (b) NA = 0.45. The laser propagation direction is given by the wavevector (\vec{k}). (c) Damage probability curves with respect to the delivered energy density (pulse energy divided by theoretical focal volume assuming linear propagation) for different focusing conditions. Estimated statistical errors are shown along Y-axis.

In Fig. 3(b), we plot the modification probability as function of the expected delivered energy density that is obtained by dividing the pulse energy by the estimated interaction volume under the assumption of linear propagation. The focal volume in which the interaction occurs is surely affected by nonlinear effects at high power. However, only a small fraction (few percent) of the

delivered energy density is absorbed for the modification threshold conditions with nanosecond laser pulses. Accordingly, we assume very localized effects making our simplified considerations valid at these threshold conditions. Therefore, we estimated the focal volume is estimated by: $V = \pi r^2 \cdot L$, where r is the beam waist and L is the length of the focus. Both of them are simulated by using the vectorial theory [26] (linear propagation) accounting for the beam overfilling conditions of the entrance pupil of objective lens. Interestingly, the results show that a similar energy density (preferably above a threshold of $\sim 0.20 \mu\text{J}/\mu\text{m}^3$) is required for achieving material modifications for all the different NA conditions. Meanwhile, we cannot derive a consistent fluence threshold (surface energy density) for modification these different NA. This tends to indicate important different phenomenon with surface modifications where the damage is usually related to the local fluence (surface energy density) reaching the target.

Another interesting observation from Fig. 3(b) is that the range of unreproducible modifications is relatively higher for lower NA cases (0.65 and 0.45) than the one for the high NA case (0.85). These results indicate that a tighter focusing is more favorable for obtaining more reproducible modifications even if nanosecond pulses are used. It is difficult to definitively conclude on the reasons behind this observation. However, one assumption can be to associate the instability with the possibility to encounter nondescript material defects which is higher when a lower NA lens is used. Another hypothesis is related to the higher pulse energy (and power) required for modification with a lower NA. This can be seen as a potential source of nonlinear transient responses of the material causing perturbations in the interactions. These include the thermal load in the interaction volume or other nonlinear propagation features, even if very modest contribution is expected from Kerr-based distortions with the considered nanosecond pulses.

3.3. Comparison with Si

The previous sections show the results of LIM inside GaAs at different pulse durations and different focusing conditions. These results show important similarities but also some interesting differences with our previous experiments of LIM of Si using a similar experimental arrangement [16]. In order to visualize that, we have presented in Fig. 4 a comparative graph on bulk damage thresholds observed in both materials.

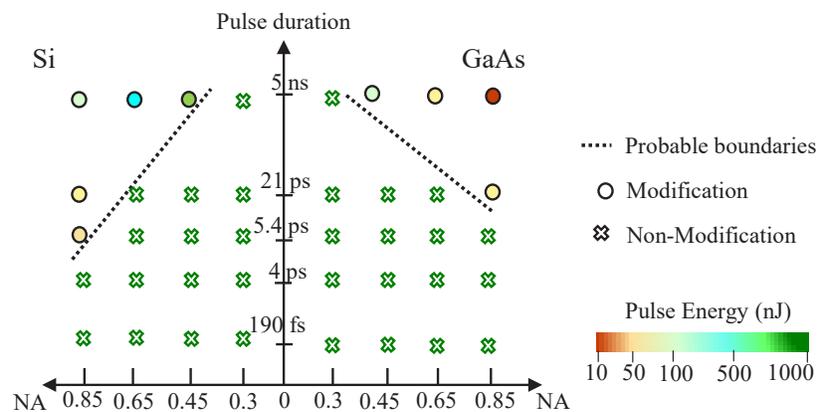


Fig. 4. A comparison of the damage threshold measured for different pulse durations and focusing conditions. Hollow circles and crosses symbolize respectively the measured thresholds and observed non-modification conditions with the available energies (up to $\approx 3 \mu\text{J}$ measured after the objective lens). The extrapolated boundaries illustrating the conditions (NA and pulse durations) for achievable modification modifiable are represented by dashed lines.

A first conclusion from the comparison is the similarity on the required conditions for pulse duration and NA to achieve modifications. For both materials, 190 fs pulses do not create any damage inside bulk even when an objective of NA = 0.85 is used. When we increase pulse duration to 21 ps, we reduce the nonlinear delocalization effects and observe bulk modification threshold conditions at NA = 0.85 at similar energy level in Si and GaAs. For nanosecond cases, we can achieve modifications in both materials in the tested range pulse energy for reduced numerical aperture down to NA = 0.45. This indicates important requirements on pulse durations and NA which applies not only for Si but also for other semiconductors like GaAs. These can be linked to important properties such as the narrow bandgaps and strong nonlinear refractive index, as shown in Table 1. Previous research have identified the delocalization of the flux due to highly efficient nonlinearities inherent to these materials and so the need to reduce the beam intensity (by NA or pulse duration increase) in the pre-focal region to achieve enough energy density for modification [10].

Beside these similarities, we also note some important differences in damage threshold values. For the 5 ns case, the threshold energy value for GaAs is nearly one order lower than that of Si case at respective NA conditions, suggesting the easiness of bulk damage of GaAs as compared to Si. This could be directly attributed to the different nature of bandgap of these two materials. As shown in Table 1, GaAs is a direct bandgap material while Si is indirect bandgap material. Accordingly, the two-photon absorption coefficient is more than 10 times higher. Then, at processing beam powers far below plasma ignition thresholds, the photon energy can be absorbed more efficiently in direct bandgap materials. This can explain why GaAs has a lower damage threshold than Si. Interestingly, we have also observed that higher pulse energy is needed to create bulk damage inside GaAs in case of 21-ps pulses as compared to 5-ns pulses. This is opposite to the case of Si and also the general case of surface modification where long pulses are usually required larger energies for breakdown. This is a direct consequence of the strong propagation effects, including filamentation, occurring during GaAs irradiation. These detrimental aspects limit the energy delivery to the focal spot, leading to apparently higher damage threshold with ultrashort pulses.

Another significant difference between Si and GaAs is the larger instability of modifications by picosecond pulses. On one hand, we observe that for GaAs, the length with NA = 0.85 can reach 37.3 μm (a standard deviation of 9.4 μm) at threshold energy for 100% modification. However, for Si, this average length is around 25 μm (standard deviation around 6 μm tested in previous work [10]) for similar conditions. On the other hand, we find that the GaAs need a pulse duration larger than 5.4 ps to obtain modification while Si can be modified with this pulse duration. These differences can be explained by the difference of nonlinear properties of these two materials. In particular, the reduced thresholds for self-focusing and for plasma formation leads to a situation inclined to filamentary effects at lower energy in GaAs in comparison to Si. The instability associated to filamentary effects leads to more irregularity and less reproducibility. At same time, a longer pulse is required to improve the energy delivery to the focal spot. While such observation is made in semiconductor, it is similar to those in some previous works concentrating in dielectrics [33,34].

4. Conclusion

In this paper, we explored the conditions of successful LIM inside an important semiconductor material, GaAs, by using different pulse durations (190 fs, 21 ps, 5 ns) and focusing conditions (NA from 0.3 to 0.85). The results indicate that there is a strong requirement on both the pulse duration and NA to achieve LIM. Longer pulses and tighter focusing are more favorable for obtaining reproducible modifications. The modification conditions are also compared to those required for LIM in Si. Except for the similarity on the general requirements on the pulse duration and NA, several differences are found between these two materials. The damage threshold for

GaAs with 5-ns pulses is typically one order of magnitude lower than for Si. At this pulse duration, free from nonlinear propagation consideration, this phenomenon is directly attributed to the direct bandgap nature of the material leading to increased laser energy absorption. In addition, the modifications induced by 21-ps pulses exhibit longer lengths and less stability for GaAs than for Si. Such features can be explained by the higher sensitivity of GaAs to self-focusing effects and subsequent filamentary features. These are important aspects which should not be ignored for the development of a reliable process technology applicable to GaAs and other semiconductors.

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Disclosures. The authors declare no conflicts of interest.

Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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