HIGHLY EFFICIENT THERMAL ABLATION OF SILICON AND ABLATION IN OTHER MATERIALS

by

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Abstract

Laser micromachining has become increasing prominent in various industries given its speed, lack of tool wear, and ability to create features on the order of micrometres. Inherent stochastic variations from thermal ablation along with detrimental heat effects, however, limit the feasibility of achieving high precision. The high number of control parameters that make laser micromachining versatile also hinders optimization due to high exploration time. The introduction of high intensity nonlinear ablation leads to more precise cuts but at a much higher, often restrictive, cost.

The work here shows that by combining an imaging technique frequently used in ophthalmology called optical coherence tomography (OCT) with a machining platform, in situ observation of ablation can be made. This combination, known as in-line coherent imaging (ICI), allows information to be gathered about the dynamics of the ablation process. Experimental results show that quality cutting of silicon can be achieved with thermal ablation and at a wavelength of 1070 nm. This result is surprising as silicon absorbs this wavelength very weakly at room temperature. It is shown here that a nonlinear thermal dependence in absorption allows a cascaded absorption effect to enable machining. With the aid of ICI, the model shown here is able to accurately predict the thermal ablation rate and help understand the ablation process. The high quality cutting achieved allows for a more cost efficient alternative to current techniques using ultraviolet diode-pumped solid state (UV DPSS) systems.
Where thermal effects such as heat-affected zones (HAZ) cannot be overcome, high intensity nonlinear ablation allows the processing of lead zirconate titanate (PZT) for high frequency arrays (used in ultrasound applications) at speeds two orders of magnitude greater than found in the literature, and potential feature sizes (< 100 µm) in polymethyl methacrylate (PMMA) unachievable by thermal ablation. The ablation mechanism here is Coulombic explosion (CE), which is a non-thermal process. Coupled with demonstrated manual and automatic feedback abilities of ICI, the processes shown here may open up new avenues for fabrication.
Thesis Contributions

The research presented here was a collaborative effort between Paul J.L. Webster (Ph.D. Candidate), Ben Y.C. Leung (former M.A.Sc. graduate), James M. Fraser (supervisor) and the author. The work here would not have been possible without the constant discussion and interaction with these colleagues.

The author was responsible for the experiments involving silicon and PZT and collected the data collaboratively with Ben and Paul for PMMA. Some optical images were obtained by Kevin D. Mortimer (Research Assistant) and Logan G. Wright (Research Assistant). The model was created by the author with collaborative discussions with James and Cole van Vlack (Ph.D. Candidate). Paul designed and assembled the ICI system and wrote the software with additions made by this author. Post processing code for imaging data was written by the author with contributions by Ben and Paul.
Acknowledgments

First, I must thank my supervisor James Fraser for his support and willingness to put up with sometimes far-fetched ideas of mine. I’m glad to have worked with you these many years and am grateful you took a chance with me as a summer student. I have learned much not only of academics, but of integrity and dedication.

My parents for their support and food. Starvation would have surely resulted without the sustenance you provided along with your unwavering faith in me.

My fellow group members, Paul Webster, Ben Leung, Elsa Xiao and Mitchell Anderson, I would not have gotten where I am without your support and ability to put up with my inane actions and antics. While I’m sure I’ve not always been the easiest person to work with, I hope at least that I have made the office more entertaining and unforgettable.

Thanks to my friends who kept me sane throughout the stressful days, of which there were thankfully few (but perhaps not as few as you would have liked). A special mention to Brian Anders who has taken the time to help edit this thesis and Jeff Grant for his keen eye when critiquing figures. I’m sure the committee thanks you as well for making it a better read.

While I recognize the need to give thanks, it is unfortunate that the acknowledgments often sound like a repeat of an Academy Award speech. However, it is the only section where I bend the rules a little, so here’s my attempt to do so:

I have often heard people remark that scientists focus too much on their rules and equations and have no imagination. I disagree, it is precisely our imagination which drives us to understand. To come up with new ideas that mostly fail, but the understanding gleaned with the possibility of success too great to pass up. Throughout history, scientists and engineers have been told to give up because something is impossible. It is impossible to fly! To go to space! To make a laser! And it is our imagination that pushes us in the face of impossibility, to succeed. So I leave you with a quote from Malcolm Reynolds, whose time with us was cut tragically short by a lack of imagination: “We have done the impossible, and that makes us mighty.”
### List of Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-line</td>
<td>Amplitude Line</td>
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<tr>
<td>B-mode</td>
<td>Brightness Mode</td>
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<td>CE</td>
<td>Coulombic Explosion</td>
</tr>
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<td>EM</td>
<td>End of Machining Laser Exposure</td>
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<td>FD-OCT</td>
<td>Fourier Domain Optical Coherence Tomography</td>
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<td>FEM</td>
<td>Finite Element Method</td>
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<td>FFT</td>
<td>Fast Fourier Transform</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width at Half Maximum</td>
</tr>
<tr>
<td>GPU</td>
<td>Graphics Processing Unit</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat-Affected Zone</td>
</tr>
<tr>
<td>ICI</td>
<td>In-line Coherent Imaging</td>
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<tr>
<td>M-mode</td>
<td>Motion Mode</td>
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<tr>
<td>NIST</td>
<td>National Institute of Standards and Technology</td>
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<tr>
<td>ND</td>
<td>Neutral Density</td>
</tr>
</tbody>
</table>
PMMA  Polymethyl Methacrylate
PSF  Point Spread Function
PZT  Lead Zirconate Titanate
OCT  Optical Coherence Tomography
QCW  Quasi-Continuous Wave
SD-OCT  Spectral Domain Optical Coherence Tomography
SFR  Spike to Flat Ratio
SNR  Signal to Noise Ratio
SS-OCT  Swept Source Optical Coherence Tomography
SW  Sidewalls
TD-OCT  Time Domain Optical Coherence Tomography
TI  Top Interface
TTL  Transistor-Transistor Logic
UV DPSS  Ultraviolet Diode-Pumped Solid State
Contents

Abstract i

Thesis Contributions iii

Acknowledgments iv

List of Abbreviations v

Contents vii

List of Tables ix

List of Figures x

Chapter 1:
   Introduction ........................................ 1

Chapter 2:
   Background ......................................... 6
   2.1 Optical absorption ................................ 7
   2.2 Properties of silicon ............................. 8
      2.2.1 Absorption coefficient ....................... 8
      2.2.2 Reflection .................................. 10
      2.2.3 Specific heat capacity ....................... 12
      2.2.4 Thermal conductivity and diffusivity ........ 13
   2.3 Modeling of laser-material interactions ......... 15
      2.3.1 FEM step sizes .............................. 18
      2.3.2 Radial thermal diffusion ..................... 21
      2.3.3 Machining beam shape ....................... 22
   2.4 Nonlinear Optics ................................ 22
   2.5 Optical coherence tomography .................... 23
   2.6 Spectral domain optical coherence tomography .. 25

Chapter 3:
   Apparatus and Experimentation .................... 29
List of Tables

2.1 Properties of silicon ................................................. 8
2.2 Properties of silicon at various temperatures ....................... 14
2.3 Optimized axial resolution ........................................... 20

3.1 Machining source data ............................................... 39
3.2 Imaging system data ................................................ 40
## List of Figures

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Band structure of silicon</td>
<td>7</td>
</tr>
<tr>
<td>2.2</td>
<td>Temperature dependence of the absorption coefficient of silicon for 1070 nm</td>
<td>10</td>
</tr>
<tr>
<td>2.3</td>
<td>Temperature dependence of the magnitude of the index of refraction of silicon</td>
<td>11</td>
</tr>
<tr>
<td>2.4</td>
<td>Temperature dependence of the specific heat capacity of silicon</td>
<td>12</td>
</tr>
<tr>
<td>2.5</td>
<td>Temperature dependence of the thermal conductivity of silicon</td>
<td>14</td>
</tr>
<tr>
<td>2.6</td>
<td>Visualization of the 1-D FEM model</td>
<td>16</td>
</tr>
<tr>
<td>2.7</td>
<td>Theoretical curve of heat distribution in 1-D</td>
<td>19</td>
</tr>
<tr>
<td>2.8</td>
<td>Chi squared values for various axial resolutions</td>
<td>20</td>
</tr>
<tr>
<td>2.9</td>
<td>Michelson interferometer setup of OCT system</td>
<td>23</td>
</tr>
<tr>
<td>2.10</td>
<td>Interferogram of OCT signal and corresponding A-line</td>
<td>27</td>
</tr>
<tr>
<td>3.1</td>
<td>Experimental apparatus</td>
<td>32</td>
</tr>
<tr>
<td>3.2</td>
<td>Gaussian fit of the imaging spectrum</td>
<td>34</td>
</tr>
<tr>
<td>3.3</td>
<td>Simulated M-mode image of a single interface</td>
<td>36</td>
</tr>
<tr>
<td>3.4</td>
<td>Side view of 3-D rendering of holes drilled in stainless steel</td>
<td>37</td>
</tr>
<tr>
<td>4.1</td>
<td>Comparison of assist gas effects in 304 stainless steel</td>
<td>42</td>
</tr>
<tr>
<td>4.2</td>
<td>Ablation of silicon with and without assist gas</td>
<td>43</td>
</tr>
<tr>
<td>4.3</td>
<td>Optical microscopy images of holes machined in silicon</td>
<td>45</td>
</tr>
<tr>
<td>4.4</td>
<td>Closeup M-mode of silicon ablation with $N_2$</td>
<td>46</td>
</tr>
<tr>
<td>4.5</td>
<td>Shapes of pulses used in the experiment and modeling simulation</td>
<td>47</td>
</tr>
<tr>
<td>4.6</td>
<td>M-mode images of ablation in silicon for varied pulse durations</td>
<td>48</td>
</tr>
<tr>
<td>4.7</td>
<td>Per pulse ablation rate as a function of pulse duration and pulse intensity</td>
<td>49</td>
</tr>
<tr>
<td>4.8</td>
<td>Simulation of temperature profile in interaction volume for 7.2 $\mu$s pulse with constant absorption</td>
<td>50</td>
</tr>
<tr>
<td>4.9</td>
<td>Simulation of temperature profile in interaction volume for 7.2 $\mu$s pulse with temperature dependent absorption</td>
<td>51</td>
</tr>
<tr>
<td>4.10</td>
<td>Temperature profiles of the interaction volume for 7.2 $\mu$s pulse</td>
<td>53</td>
</tr>
<tr>
<td>4.11</td>
<td>Temperature profiles of the interaction volume for 38.5 $\mu$s pulse</td>
<td>53</td>
</tr>
<tr>
<td>4.12</td>
<td>Experimental and simulated ablation depths per pulse</td>
<td>54</td>
</tr>
<tr>
<td>4.13</td>
<td>Reduced chi squared values for varying $L_{Radial}$</td>
<td>55</td>
</tr>
<tr>
<td>4.14</td>
<td>Ablation depth per pulse for simulated top hat pulses with same pulse energy</td>
<td>56</td>
</tr>
<tr>
<td>Section</td>
<td>Description</td>
<td>Page</td>
</tr>
<tr>
<td>---------</td>
<td>-----------------------------------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>4.15</td>
<td>Ablation depth per pulse for varying spike heights with same total pulse energy</td>
<td>57</td>
</tr>
<tr>
<td>4.16</td>
<td>Ablation depth per pulse for varying spike heights as a function of energy</td>
<td>58</td>
</tr>
<tr>
<td>4.17</td>
<td>M-mode of silicon ablation with 9 ps pulses</td>
<td>61</td>
</tr>
<tr>
<td>4.18</td>
<td>Optical microscopy image of nonlinearly ablated hole in silicon</td>
<td>62</td>
</tr>
<tr>
<td>4.19</td>
<td>Ultrafast ablation of silicon imaged by the 1320 nm OCT system</td>
<td>62</td>
</tr>
<tr>
<td>5.1</td>
<td>M-mode of nonlinear ablation of PZT</td>
<td>64</td>
</tr>
<tr>
<td>5.2</td>
<td>Top view of optical microscopy image of nonlinearly ablated hole in PZT</td>
<td>65</td>
</tr>
<tr>
<td>5.3</td>
<td>Two sets of four trenches cut in PZT</td>
<td>66</td>
</tr>
<tr>
<td>5.4</td>
<td>Square feature cut in PMMA</td>
<td>68</td>
</tr>
<tr>
<td>5.5</td>
<td>Optical microscopy image perpendicular trenches cut in PMMA</td>
<td>69</td>
</tr>
<tr>
<td>5.6</td>
<td>Side view of holes drilled in stainless steel with and without feedback</td>
<td>70</td>
</tr>
</tbody>
</table>
Chapter 1

Introduction

The advent of the first working laser in 1960 was met with skepticism, with critics decrying it as a ‘solution looking for a problem.’ The notion was very quickly overturned and it is now difficult to imagine a world without this technology. Micro-machining is one area where the lack of tool wear and precision provided by lasers makes them a natural fit. Rapid growth in this industry has pushed a need for more effective and cost-efficient techniques while simultaneously improving upon the precision [1]. Indeed, successes with ultrafast technology (<10 ps) show that cutting with extreme precision and without the negative attributes of thermal ablation, such as heat-affected zones (HAZ) and microcracking, can be achieved [2, 3]. However, these systems are often cost prohibitive and the dynamics of thermal ablation is left largely unexplored in favour of investigating these more nonlinear time regimes. Recent literature suggests that given the right parameters, nonlinear effects in thermal ablation can also lead to favourable results as well as the ability to ablate using unconventional wavelengths with respect to the material [4]. Even given the highly controllable nature of laser systems and improvements in empirical models [5–8], results (e.g. ablation rates) continue to be highly varied due to the stochastic nature of the ablation process.
despite repeated trials. Along with the high number of optimization parameters, full exploration is often time consuming and economically prohibitive. These parameters include repetition rate, pulse energy, pulse duration, pulse shape, peak power, focal range, focal size, and ablation environment (assist gas, pressure, etc.).

Micromachining of silicon is a growing industry due to its many applications in microelectronics and solar panel technology. For thin wafer dicing, the process has traditionally been done with a precision mechanical saw [9]. Aside from the lack of tool wear, laser machining would also be able to create thinner slices than mechanically possible, allowing more chips to be created on a single wafer [9] as well as faster processing speeds, both of which have been demonstrated recently [10]. With the development of ultraviolet diode-pumped solid state (UV DPSS) lasers, industry has begun to move away from mechanical processing techniques for silicon. Recent work with these systems has shown that high quality (e.g. reduced HAZs, no microcracking) processing is possible with limited thermal effects once parameters have been optimized [11]. However, fibre lasers are easier to integrate into existing machining platforms due to their non-free-space nature and are more cost effective compared with the UV optics required for UV DPSS systems. Recent results have shown that indeed this is a new possible area of development as nanosecond fibre lasers have been demonstrated to cut well in silicon [4, 12]. What is curious and warrants further investigation is the underlying mechanisms that allow thermal ablation for the infrared wavelengths of fibre lasers, wavelengths at which silicon has a long absorption depth. Understanding of this phenomenon could lead to additional improvements in the machining process. Given the stochastic nature of the process, ex situ imaging techniques only provide a portion of the whole picture and so both ex and in situ imaging are required.

Current non-contact ex situ imaging modalities include optical microscopy and
electron microscopy, both of which can obtain high resolution ($<1 \mu m$) on the feature. These techniques provide excellent 2-D information and are well suited to imaging at or near the surface of the material. Profilometry can also be applied to obtain depth information given a low aspect ratio feature. Given higher aspect ratios, profilometry is no longer viable (due to the size of the tip being unable to fit into the feature for the diameter of the holes presented here) and a side-polishing technique is required. This is a slow process and is further limited by the number of features that need to be created and measured to determine repeatability for a single set of parameters. The side-polishing process involves physically grinding the sample to obtain a side profile view, requiring the destruction of part of the sample and can alter the shape of the feature under study. Also, the ex situ nature of these techniques does not allow a study of ablation dynamics and cannot provide feedback, ensuring that ideal parameters are still affected by the stochasticity of the process.

Work has been done by various groups to provide an in situ imaging solution. A Linnik interferometry technique has shown that nanometre depth resolution and femtosecond temporal resolution can be achieved [13]. This and other interferometry techniques [14], however, obtain their high resolution from the phase-sensitive nature of the measurements and thus are limited in their abilities to measure features deeper than the wavelength of the source imaging light. Such deeper features would add ambiguity to their results, thus limiting the applications these methods can be applied to shallow features. Another approach is the use of high-speed photography to image holes drilled near the edge of a material where measurements are made of the deformation along the edge [15]. While high aspect ratio holes can be imaged with this method, it requires extrapolation to determine the hole depth and has a limited resolution in the axial direction. Limitations again arise as the feature must be created near the edge of the material. Time-gated imaging of back-scattered light
has also been used with micrometre resolution, but limited to a 1 kHz imaging rate due to their source [16]. As industrial applications often require much higher machining rates (5 kHz to > 100 kHz process depending), this results in thermal effects on similar time scales and are thus too fast to be explored [17].

A solution arises in optical coherence tomography (OCT), a technique that has found widespread use in ophthalmology [18, 19]. OCT can provide depth resolutions of up to 2.1 µm [20] and sub-micron transverse resolution [21] allowing the investigation of a wide range of feature sizes. The two resolutions are decoupled and the optical nature of this technique makes it an ideal candidate to combine with a laser machining platform. The technique also obtains information from multiple depths and as will be shown later, allows the study of features below the ablation front in silicon. When the imaging beam is raster scanned, OCT is able to create 2-D and 3-D images of the feature, both ex and in situ, without the need to move the sample. This has been demonstrated in the ablation of biological samples [22–25]. By removing the raster scanning and fixing the imaging beam in-line with the feature, the imaging line rate can be greatly improved to over 300 kHz with a temporal resolution of 1.5 µs [26]. This combination of OCT and micromachining, known as in-line coherent imaging (ICI), has allowed us to show previously unseen dynamics in the thermal ablation of silicon and stainless steel, and provides an opportunity to improve the accuracy of industrial processes via active feedback [27, 28], as will be discussed in this thesis.

As well, the thermal ablation of silicon will be investigated, specifically the mechanisms that drive the ablation process when a wavelength of 1070 nm is used for the machining beam. This is a surprising result as silicon is highly transmissive at this wavelength with an absorption depth of ∼1.3 mm [29]. This result has been confirmed in pulse time regimes varying from micro- to nanoseconds [4, 26, 30]. The ablation process takes advantage of a nonlinear thermal effect that is inherent in silicon and
provides a driving mechanism that enables cutting. A custom 1-D finite element method (FEM) model is created to predict and elucidate the pulse interaction with the silicon and does so accurately. ICI is used to verify the model as well as provide additional information on the dynamics of cutting, allowing a comparison between thermal ablation and nonlinear ablation, which is explored in Chapter 4. However, thermal ablation has its limitations, which can be overcome with nonlinear ablation. Due to high peak irradiances, nonlinear ablation allow for laser micromachining to improve upon current fabrication techniques (thermal ablation, etc.) for high frequency transducers in fine grain ceramic and microfluidic channels in polymethyl methacrylate (PMMA) [31, 32]. The processing of these materials and the ability to apply feedback with ICI is shown in Chapter 5. The ability to machine silicon in the infrared with a fibre laser provides a more economical alternative to current UV DPSS systems. This, coupled with the ability to provide active feedback via ICI, opens new avenues for future developments in industrial laser micromachining.
Chapter 2

Background - Properties of Silicon, Numerical Modeling, Nonlinear Optics, and Imaging Technique

To accurately model the change in temperature in silicon due to the machining pulse interaction, several nonlinear properties of silicon must be taken into account. When these properties are coupled with additional information obtained from OCT imaging (i.e. ablation rate, relaxation time of melt) an accurate model can be made to help further unravel the underlying mechanisms as well as provide a platform to predict the interaction with simulated pulses. The in situ nature of OCT has shown that other explanations in the literature do not accurately reflect the ablation process observed here. This chapter will help to elucidate the thermally dependent properties in silicon important to ablation, the modeling method and its limitations, the applicable theory of nonlinear optics (specifically high intensity ablation), as well as the theory of OCT and the specific subset chosen for ICI.
2.1 Optical absorption

Figure 2.1: Band structure of silicon. The indirect band gap for a 1.1 eV photon shows the need for coupling to a phonon with a large momentum [33].

One of the goals of the work presented here is to allow for the drilling of high aspect ratio features on the micrometre scale. Laser micromachining is a solution with processing currently done with UV DPSS lasers. Absorption of the pulse leads to electrons promoted into the conduction band where their eventual decay will transfer energy to phonons and heat the system. Fig. 2.1 shows the band structure of silicon and it can be seen that UV lasers (> 3 eV) have a direct interband absorption.

UV DPSS lasers are more expensive than their fibre laser cousins at wavelengths of around one micron, leading to the question of whether similar cutting can be achieved. Fig. 2.1 shows that for a wavelength of 1070 nm (1.16 eV), the photon has enough energy to allow for an indirect transition only if it can couple with a phonon. This leads to very weak absorption suggesting that much higher fluences are required to
achieve the same temperature rises as UV systems. However, temperature changes cause other properties of silicon to change, which must be taken into account for accurate modeling. These effects with rising temperature are explored in the next section.

### 2.2 Properties of silicon

Table 2.1 shows some of the important properties of silicon used in the model [34].

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Temperature (K)</td>
<td>1687</td>
</tr>
<tr>
<td>Vaporization Temperature (K)</td>
<td>3538</td>
</tr>
<tr>
<td>Heat of Fusion (J/g)</td>
<td>1788</td>
</tr>
<tr>
<td>Heat of Vaporization (J/g)</td>
<td>12782</td>
</tr>
</tbody>
</table>

There are also several properties of silicon that are found to be highly dependent on temperature and are included in the modeling. Two processes work to aid the heating of silicon (absorption coefficient and thermal conductivity/diffusivity) and two help to slow down the process (reflection and specific heat capacity). Thus, there is an interplay between these properties that must be taken into account for an accurate model. As well, the model will be able to show which thermal dependency is the dominant effect.

#### 2.2.1 Absorption coefficient

An important mechanism responsible for infrared thermal ablation of silicon is the nonlinear thermal dependence of its absorption coefficient. Assuming a constant absorption depth at room temperature (1 mm in silicon for a wavelength of 1070 nm), this leads to a temperature increase of only 336 K given a 560 $\mu$J pulse (used in the experiments) when the model is applied (see Section 4.3). This is far below
the melting temperature of 1687 K, the minimum temperature required for thermal material removal in silicon [34]. However, the absorption coefficient is instead found to depend strongly upon temperature, \( T \), due to several mechanisms.

First, as temperature increases, the band gap decreases allowing the incoming beam access to a higher density of electrons and holes in the valence and conduction band respectively. This has been characterized by Bludau et al. [35] for temperatures from 0-300 K and Li et al. [36] for temperatures from 0 - 490 K, where the trend continues for higher temperatures, but nonlinearly.

As well, there is an increase in the density of free carriers due to rising temperature; this results in the free carrier absorption becoming a much more noticeable absorption mechanism compared with the interband absorption. The free carrier absorption cross-section, \( \sigma \), is found to increase linearly with temperature as

\[
\sigma = 1.7 \times 10^{-20} \cdot T
\]

in units of cm\(^2\). This relationship is consistent with absorption due to acoustic phonon scattering [37]. Thus, a combination of these mechanisms leads to the rising absorption and the cascaded absorption effect that enables rapid temperature rise (expanded upon in Section 4.3).

Therefore from experimental results [38, 39], the optical absorption, \( \alpha \), is governed by

\[
\alpha = \left[ \frac{T}{172.3} \right]^{4.25}
\]

for a wavelength of 1064 nm, where \( \alpha \) is in units of cm\(^{-1}\). Eqn. 2.2 is true for a temperature range of 100<\(T<1687\) K and Fig. 2.2 shows this relationship. The absorption depth is the inverse of the absorption coefficient (i.e. \( \alpha^{-1} \)). So given a
rise of 336 K from room temperature, the absorption depth will have decreased from 1 mm to 41 \( \mu \text{m} \), dramatically increasing the total amount of absorbed energy as well as depositing the energy into earlier depths. This results in extreme heating of the surface. Once the silicon reaches the melting temperature, the absorption depth is assumed to remain constant at 0.6 \( \mu \text{m} \) (absorption depth at the melting temperature calculated from Eqn. 2.2).

Other models in the literature also make note of a temperature dependent absorption [12], but make the simplification of a constant absorption corresponding to a higher temperature in their model. Section 4.3 will show that this is not an accurate assumption.

### 2.2.2 Reflection

As the temperature rises in solid silicon, it has been found that there is also a noticeable change in the magnitude of the index of refraction, \( n \) [36, 40]. Fitting to data
from [36] for a wavelength 1070 nm, it is found that

\[ n = 2.6 \times 10^{-7}T^2 + 1.6 \times 10^{-4}T + 3.5 \]  

(2.3)

for a temperature range of 100 - 800 K. The trend is extrapolated and assumed to continue up to the melting temperature, in agreement with the literature (discussed below).

![Figure 2.3: Temperature dependence of the magnitude of the index of refraction of silicon for a wavelength of 1070 nm.](image)

This leads to a significant change in the reflection from 31% at room temperature to 41% at the melting temperature, which is close to recent work showing a reflection of about 39% before liquefaction [41]. This temperature dependence in the magnitude of the index of refraction is shown in Fig. 2.3.

An even more significant change occurs once the silicon liquefies where recent work has shown a reflection of 70% [41]. This greatly reduces the amount of energy from the pulse that enters the silicon.
2.2.3 Specific heat capacity

Specific heat capacity, $C_p$, of silicon also rises with temperature by almost 50% from room to the melting temperature. Once the silicon liquefies, the specific heat maintains a constant value [42]. Fitting to data found in [42], this relationship is described by

$$C_p = 1.3[\ln(T)]^3 - 27.3[\ln(T)]^2 + 190.2[\ln(T)] - 423.9$$  \hspace{1cm} (2.4)

where $C_p$ is in units of $\frac{J}{mol\cdot K}$. This shows that silicon requires increasingly more energy for a given rise in temperature at higher temperatures. This equation is valid from 200 - 1687 K. Fig. 2.4 shows the temperature dependence of the specific heat capacity.

Figure 2.4: Temperature dependence of the specific heat capacity of silicon.
2.2.4 Thermal conductivity and diffusivity

For the temperature range of 300 - 1687 K, the thermal conductivity, $k_C$, decreases with increasing temperature in solid silicon [39, 43]. Once the silicon turns into liquid, there is an abrupt jump followed by a steady increase for rising temperatures of liquid silicon.

In solid silicon, the thermal conductivity can be separated into two temperature regimes: $0.5\theta < T < 1.6\theta$ and $T > 1.6\theta$, where $\theta = 674$ K is the Debye temperature (point at which the Debye model no longer applies for higher temperatures) of silicon [44]. These relationships are given by

$$k_C = \begin{cases} 
\frac{1521}{T^{1.226}} & 300 K \leq T \leq 1200 K \\
\frac{8.96}{T^{0.302}} & 1200 K \leq T \leq 1687 K 
\end{cases}$$

and shows a dramatic decrease in thermal conductivity as the melting temperature is approached ($k_C$ is in units of $\text{W/cm/K}$) and was determined through experimental data from [39, 43]. Fig. 2.5 shows this relationship. For the first regime, it has been suggested that the higher order phonon scattering effects increase the thermal resistivity. In the second regime, there is now a major contribution and competing effect from the electronic thermal conductivity, where electron-hole pairs with the band gap energy diffuses down the temperature gradient [44].

Once the silicon liquefies, there is a jump in thermal diffusivity, $D_T$, with a continued rise of approximately 0.1%/K, which gives

$$D_T = 0.26 \times 10^{-4} \cdot (1.00083)^{T-1687}$$

with units of m$^2$/s and is related to thermal conductivity by $D_T = \frac{k_C}{\rho C_P}$, where $\rho$ is the density of silicon. This relationship is determined through experimental data by
The rise is attributed to a sudden increase in the density of free electrons, which are the dominant carriers of heat in liquid silicon.

It can be seen that there is an interplay of the four aforementioned properties that govern how the temperature rises. Table 2.2 shows how these values change from room temperature to the melting point of silicon at 1687 K [34].

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Absorption (cm$^{-1}$)</th>
<th>Refection (%)</th>
<th>Specific Heat Capacity ($\frac{J}{mol\cdot K}$)</th>
<th>Thermal Conductivity ($\frac{W}{cm\cdot K}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{Room}, 293$</td>
<td>9.5</td>
<td>31</td>
<td>19.8</td>
<td>1.4</td>
</tr>
<tr>
<td>$\lim(T \rightarrow 1687)$</td>
<td>$1.6 \times 10^4$</td>
<td>41</td>
<td>29.0</td>
<td>0.2</td>
</tr>
<tr>
<td>$T &lt; 1687$</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1687</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$Liquid\ Silicon,$</td>
<td>1.6$\times 10^4$</td>
<td></td>
<td>27.2</td>
<td>0.7</td>
</tr>
</tbody>
</table>

While all the temperature dependent changes are important and considered in the model, it is clear that the decrease in absorption depth by three orders of magnitude is the most dramatic change of the four temperature dependent properties.
2.3 Modeling of laser-material interactions

From the results of the experiments (see Chapter 4) it was clear that ablation occurs within the first pulse of the machining beam. A model describing the intrapulse effects will allow a better understanding of the ablation process, a determination of the dominant contributions, as well as a way to predict optimal pulse shapes for improved machining (i.e. increased material removal rates). A purely analytical model is created by Hendow et al. [12], but they make the assumption of a constant absorption. Section 4.3 will show that this assumption is not valid for the model presented here.

The model here uses a 1-D finite element method (FEM) and examines the interaction of a defined volume with an incoming laser pulse. The volume is considered to be cylindrical where the 1-D FEM takes place along the direction of laser propagation (z-direction). The 1-D model is able to take into account 3-D effects analytically, which greatly improves the computational time over traditionally 2-D or 3-D FEMs as well as providing a much simpler model. Fig. 2.6 is a visualization of the model.

At every time step, a portion of the incoming beam is absorbed by the interaction volume resulting in a temperature rise. As well, radial (between the slice and the bulk sample) and axial (between slices) thermal diffusion takes place given a difference in temperature. The temperature change and thermal diffusion are calculated for every slice and time step.

The energy delivered to each slice from the machining beam for a given time, \( UO_{z,t} \), is

\[
UO_{1,t} = I_t \cdot \delta t \cdot (1 - Reflect) \cdot (1 - e^{-\alpha \cdot \delta t})
\]  

(2.7)
Figure 2.6: Visualization of the 1-D FEM model.

\[ UO_{z,t} = (I_t \cdot \delta t \cdot (1 - \text{Reflec}) - \sum_{i=1}^{z-1} U_{z,i}) \cdot (1 - e^{-\alpha \cdot \delta l}) \]  
(2.8)

where \( z \) is an index for the slice (i.e. \( z=1 \) is the top most slice), \( t \) is an index for a specific time, \( I_t \) is the intensity of the pulse at a specific time index, \( \delta t \) the temporal resolution (i.e. temporal step size) of the model, \( \text{Reflec} \) is the percentage of pulse that is reflected, and \( \delta l \) is the thickness of each slice. Thus \( I_t \cdot \delta t \) gives the energy delivered by the pulse at a specific time step (intensity of the pulse at a specific time step integrated over the length of the time step).

Axial thermal diffusion is given by

\[ UZ_{z,t} = k_c \cdot \frac{A_{TH}}{\delta l} \cdot \delta t \cdot (T_{z,t} - T_{z+1,t}) \]  
(2.9)

where \( UZ_{z,t} \) is the energy transferred from slice \( z \) to slice \( z + 1 \), \( T_{z,t} \) the temperature in slice \( z \), and \( A_{TH} \) the surface area of the slice perpendicular to the \( z \)-direction (this
is defined in Section 2.3.3). The entire interaction volume, $V$, can then be defined as $V = A_{TH} \cdot \delta l \cdot N$ where $N$ is the total number of slices. Note that $UZ_{z,t}$ can be both negative and positive depending on the temperature gradient.

The bulk silicon is assumed to heat up as well with $L_{Radial}$ defined as the radial distance between the interaction volume and the position in the bulk silicon that is at room temperature. From $L_{Radial}$, the amount of energy, $UR_{z,t}$, that diffuses radially out of slice $z$ can be calculated. These parameters are defined and discussed in greater detail in Section 2.3.2.

The total energy absorbed by a slice, $UA_{z,t}$, for a given time and step index is

$$UA_{z,t} = UO_{z,t} + UZ_{z+1,t} - UZ_{z,t} - UR_{z,t}$$ (2.10)

which gives a temperature rise, $\Delta T_{z,t}$, for a given slice as

$$\Delta T_{z,t} = \frac{UA_{z,t}}{C_p \cdot m}$$ (2.11)

and a new temperature of

$$T_{z,t+1} = T_{z,t} + \Delta T_{z,t}$$ (2.12)

Here, $C_p$ is in units of $\frac{J}{g \cdot K}$, and $m$ is the mass of each slice. This is the general case when the slice is completely a solid or a liquid. Heat of fusion and vaporization is taken into account when a slice undergoes phase change.

Given the 1-D nature of the model, important things to consider are the thickness of each slice (i.e. model resolution), $\delta l$, as well as the time step size (i.e. temporal resolution), $\delta t$. These two step sizes are found to be dependent on each other and are discussed in the next section. As well, only axial thermal diffusion is calculated via FEM while the radial thermal diffusion is taken account analytically, which extends
this 1-D FEM to account for 3-D effects. This allows for a much simpler model with faster computation times. The radial thermal diffusion is independent of the FEM step size calculations and discussed in Section 2.3.2.

2.3.1 FEM step sizes

For the model, the pulse energies and shapes for the experiment were measured and used as the input energy, which allows a more accurate calculation to compare with experimental results as opposed to purely simulated pulses. The time steps were chosen before the z-step sizes to sufficiently take into account the optical and temperature dependency effects. To test for convergence, finer time steps by an order of magnitude were used and showed no change in final results outside of discretization uncertainties and much greater computation time. While the axial resolution in the model can be set to an arbitrary fineness (at the cost of calculation time), an issue arises when this resolution becomes too small for the predetermined temporal resolution. Other people have observed in modeling electrodynamics that there are specific bounds on the lattice space increments for a given time step. Outside of these bounds, numerical instability occurs causing results to increase without limit [46]. This is known as numerical dispersion and a similar effect is present here.

Thus, to ensure the highest possible resolution without numerical instability, the model is compared to a 1-D analytical case. Beginning with the heat diffusion equation,

$$\nabla^2 u = \frac{1}{D_T} \frac{\partial u}{\partial t}$$  \hspace{1cm} (2.13)

where $u$ is the temperature as a function of time and space, a temperature gradient can be solved with initial conditions. Following a similar example from [47], Eqn. 2.13
CHAPTER 2. BACKGROUND

Figure 2.7: Theoretical curve of heat distribution in 1-D. This is the solution to Eqn. 2.14 for a time of 8 µs.

is solved for the specific parameters of the experiment. Given heat diffusion in 1-D and the following initial conditions: a) a linear gradient from $T_{\text{Room}}$ to 1686 K at $t \leq 0$ from $z = 0$ to $z = l$ respectively, and b) $u(0, t) = u(l, t) = T_{\text{Room}}$ for $t \geq 0$, where $l$ is the total height of the interaction volume, the solution becomes

$$u(z, t) = \frac{2 \cdot (1686 - 293.15)}{\pi} \sum_{n} \frac{(-1)^{n+1}}{n} \sin\left(\frac{n \cdot \pi}{l} z\right) e^{-\left(\frac{n \cdot \pi}{l}^2 D_T \cdot t\right)}$$

(2.14)

This can be solved for specific points in time and compared with modeling results. Fig. 2.7 shows the temperature distribution corresponding to $t = 8$ µs, a height $l = 270$ µm, and a constant $D_T = 9.6 \times 10^{-6}$ m²/s corresponding to silicon at a temperature of 1686 K. For comparison, temperature dependencies of thermal conductivity and diffusivity in the model are deactivated and no energy enters the system (i.e. no pulse).

Fig. 2.8 shows the chi square values when comparing the model to theory for varying axial resolutions and a time resolution of 0.8 ns. This clearly shows a range
Figure 2.8: Chi squared values for various axial resolutions. The comparison is done for a temporal resolution of 0.8 ns. The highest axial resolution used is when chi squared is at a minimum (i.e. lowest variation from theory). Noting the logarithmic nature of the plot, numerical instability causes rapid deviation from theory outside set bounds.

of values that can be used where there is numerical stability, and a highest axial resolution before numerical instability sets in. Numerical dispersion for heat transfer occurs as too small a step size (for a given time resolution) limits the rate of heat transfer whereas a large step size leads to averaging and discretization uncertainty. Three time steps were used in the experiments (0.8 ns, 2 ns, and 4 ns) and the optimization was done for all three.

Table 2.3 shows the highest axial resolution for a given time step as determined from the optimization process (resolution corresponding to the lowest chi squared for a time step).

<table>
<thead>
<tr>
<th>Temporal Resolution (ns)</th>
<th>0.8</th>
<th>2</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Highest Axial Resolution (µm)</td>
<td>0.125</td>
<td>0.2</td>
<td>0.28</td>
</tr>
</tbody>
</table>
2.3.2 Radial thermal diffusion

To improve computational time and provide a simpler model over 2-D or 3-D FEM, a 1-D FEM model was created with steps discretized in the direction of propagation of the machining and imaging beams (this is also known as the axial or z-direction). Cylindrical symmetry was assumed, matching the shape of the experimental holes. To extend this model to three dimensions radial heat diffusion is taken into account. This is the dominant 3-D effect and an analytical solution for a similar geometry to the one described here is given by [48, 49]. With the slice at a specific temperature, $T_{z,t}$, at $r = 0$ (where $r$ is the radial direction) and room temperature at $r = L_{Radial}$, [48, 49] gives the solution for the temperature gradient as a first order Bessel function with $r = 0$ as the peak of the Bessel function, and $r = L_{Radial}$ the first zero crossing ($\alpha_0$) of the Bessel function. This gives a time constant, $\tau$, which is the time it takes for the difference in temperature ($T_z - T_{Room}$) to drop to $1/e$ and is defined by

$$\tau = D_T \cdot \left( \frac{\alpha_0}{L_{Radial}} \right)^2$$  \hspace{1cm} (2.15)

where $L_{Radial}$ is the physical distance from the volume slice to room temperature in the bulk sample. This gives the energy diffusing radially out of each FEM slice for a given time step, as

$$UR_{z,t} = C_p \cdot m \cdot (T_{z,t} - T_{Room}) \cdot (1 - e^{-\delta t/\tau})$$  \hspace{1cm} (2.16)

Here, $C_p$ is in units of $\frac{J}{g \cdot K}$. A constant $L_{Radial}$ is a source of uncertainty in the modeling as $L_{Radial}$ would vary with increasing temperature (the bulk sample also heats up). However, this would require changing the model to a 2-D FEM, significantly increasing the computation time and so is assumed to be constant. As a first approximation, the temperature in the radial direction can be assumed to drop to room temperature
on the order of the machining spot size (typically on the order of 10 µm). A better approach given the experimental data is to use $L_{\text{Radial}}$ as a fitting parameter. The assumption of a constant $L_{\text{Radial}}$ is shown to be acceptable in Section 4.3 where simulation and experimental results are compared.

### 2.3.3 Machining beam shape

The machining beam has a Gaussian energy distribution in real space. An assumption is made that a top hat equivalent area, $A_{\text{TH}}$, is the area over which all the energy in the beam interacts with the sample for the model. Thus the top hat pulse has the same total energy as well as the same peak intensity as the Gaussian pulse. This is a standard approximation [50] and is defined by

$$A_{\text{TH}} = \frac{\pi \cdot (w/2)^2}{2}$$

where $w$ is the FWHM of the Gaussian beam. $A_{\text{TH}}$ is compared with the experimental hole size in Section 4.1.

### 2.4 Nonlinear Optics

In general, when the intensity is high enough, the pulse is able to induce self focusing in air, second harmonic generation, sum and difference frequency generation, etc. depending on certain conditions. For high intensity nonlinear ablation (the experiments described in this thesis), the beam is able to remove a large number of electrons from their orbitals creating a repulsive charged cloud, which drives material removal. This is known as Coulombic explosion (CE) and is the main mechanism for nonlinear ablation [2]. This allows for much cleaner and deterministic cutting without the detrimental effects of thermal ablation [2].
There are two regimes to high intensity nonlinear ablation: a high quality (i.e. no surface damage, no surface deposits) cutting regime with low pulse energy and slow ablation rates, and a lower quality cutting regime with higher pulse energies and faster ablation rates [51]. The lower quality regime is of importance to industry as it allows for much higher throughput in terms of material processing and involve fluences of $>10 \text{ J/cm}^2$ [51].

2.5 Optical coherence tomography

![Michelson interferometer setup of OCT system.](image)

Figure 2.9: Michelson interferometer setup of OCT system.

To aid in the modeling and understanding of melt effects, a technique known as OCT is used to monitor ablation in situ. The most basic OCT system uses a Michelson interferometer with a low temporal coherence light source; the back-scattered light from the sample is used to determine the depth with respect to a known reference path length (Fig. 2.9). The first incarnation made use of a moving reference arm to image the sample at various depths [52]. This is known as Time Domain OCT (TD-OCT).
This setup allowed for a large field of view limited only by the travel distance of the reference mirror. However, the mechanical requirement of the reference arm severely limits the acquisition rates that are available, with one of the highest reported speeds of only 13 kHz [53]. This shows that TD-OCT would be unsuitable for the study of the faster dynamics happening within the ablation process.

Since the development of TD-OCT, research has begun to shift towards Fourier Domain OCT (FD-OCT), which removes the need for a moving reference arm at the cost of a more expensive detector system and a smaller imaging dynamic range due to higher bit depth detectors used in TD-OCT. In FD-OCT, the frequency of the interference pattern is measured and depth is obtained through a Fourier transform. The pattern can be collected temporally, known as Swept Source OCT (SS-OCT), or spatially, known as Spectral Domain OCT (SD-OCT).

In SS-OCT, a wavelength-tunable laser source is used to scan through a set bandwidth [54]. The laser emits a very narrow linewidth and the spectral interferogram is recorded with a photodiode synchronized to the source. This allows for much faster scanning speeds than a mechanical reference arm. In fact, the fastest line rates published for OCT systems use the swept source design with rates of up to 20 MHz [55]. However, the expensive sources required for such systems produce an economical challenge for industrial applications. Also, an interesting motion artefact arises when the ablation front or any layer moves by a distance, $\Delta z$, within an axial line rate. This artefact results in a broadening of the signal defined by

$$\Delta z_{\text{max}} = \frac{\delta z \cdot (M^2 - 1)^{1/2}}{2\eta},$$

where $M$ is the tolerated broadening factor, $\delta z$ is the full width at half maximum (FWHM) axial resolution, and $\eta$ is a factor determined by the FWHM of the spectrum.
Thus, given sufficiently fast recast (i.e. material ejected from the drilled holes which settles on the surface) and/or ablation front, the signal would be too broadened to be of value.

For SD-OCT, instead of a tunable laser, a broadband source is used instead. With the aid of a diffraction grating, the spectral interferogram is measured with a linear array. The noise found in SD-OCT is reduced as it is averaged over many pixels compared with SS-OCT while maintaining a higher axial line rate than TD-OCT. The line rate is now limited by camera technology with rates of $>300$ kHz for silicon detectors [26, 28] and $>91$ kHz for InGaAs detectors [58]. The same motion artefact occurs with SD-OCT as with SS-OCT, except now the linear array averages this signal, which causes a reduction or disappearance of the moving interface from the final image. This is known as fringe washout and the signal to noise ratio (SNR) penalty is given by

$$SNR_{Penalty} = \frac{\sin^2(k_0 \Delta z)}{(k_0 \Delta z)^2},$$

where $k = 2\pi/\lambda$, $\lambda$ is the wavelength, and $k_0$ is the wavenumber corresponding to center wavelength, $\lambda_0$ of the imaging beam [57]. Given the high sensitivity nature of FD-OCT in general, this becomes a smaller issue than in SS-OCT where at worst the motion removes itself from the image rather than distorting the remaining data. Thus, with the advantages discussed here, SD-OCT is used to conduct in situ monitoring in this work and in ICI.

2.6 Spectral domain optical coherence tomography

For the simplified case of a single mirror at the reference and sample arms (see Fig. 2.9) with the assumption that the imaging beam is at a single frequency, the resulting
electric field as seen by the detector can be described by

\[
E_{\text{det}} = E_{\text{sam}} + E_{\text{ref}} = A_{\text{sam}} e^{i(\omega t - kz_{\text{sam}})} + A_{\text{ref}} e^{i(\omega t - kz_{\text{ref}})}
\]  
(2.20)

where \( E \) is the electric field, \( A \) is the amplitude of the signals, \( z \) the path length of the arms, \( \omega \) the frequency of the source, and the subscripts \( \text{det}, \text{sam}, \text{and ref} \) corresponding to the values associated with the detector, sample arm, and reference arm respectively [59].

However, it is the intensity, not electric field, that is of interest and read by the detector, given as

\[
I = |E_{\text{det}}|^2 = A_{\text{sam}}^2 + A_{\text{ref}}^2 + 2A_{\text{sam}}A_{\text{ref}} \cos(k \cdot \Delta l)
\]  
(2.21)

where \( \Delta l = z_{\text{sam}} - z_{\text{ref}} \) is the path length difference between the two arms [59]. The first two terms in Eqn. 2.21 represents the intensities of the sample and reference arms, which are DC terms that are measured a priori and subtracted from the final image. The third term contains the interference information and it is this term that gives rise to the depth information when a Fourier transform is taken. Fig. 2.10 shows an artificial interference pattern corresponding to one interface at the sample as well as the output after a Fast Fourier Transform (FFT). This output represents one amplitude line (A-line), a collection of which gives the final image. The FWHM of the peak is the point spread function (PSF), which gives the axial resolution. It should be noted that the FFT gives rise to two peaks (as seen in Fig. 2.10) as expected due to the complex nature of the FFT. In OCT this is known as complex conjugate ambiguity and as a result, half of the signal is removed from the final image. This is
Figure 2.10: Simulated signal of the interferometric term for a single interface assuming a Gaussian imaging beam. a) The interferogram in k-space corresponding to a center wavelength for an 805 nm source and b) the image after FFT processing (A-line) showing the artificial interface at 300 µm, which is the optical path length distance of the two arms. The two peaks are due to the complex conjugate ambiguity. This figure is adapted from [60].

justified as during experimentation, all imaging is done on one side (specifically the positive side) of zero delay.

Again assuming a Gaussian imaging beam, the axial resolution of the system is

\[ \delta z = \frac{2 \ln 2}{\pi} \frac{\lambda_0^2}{\Delta \lambda}, \quad (2.22) \]

where \( \Delta \lambda \) is the spectral width of the imaging source [61, 62]. As previously mentioned, the transverse resolution, \( \Delta x \), is decoupled from the axial resolution and defined by the \( 1/e \) diameter for a Gaussian beam. This is determined by the focusing optic and represents the spot size of the focused beam at the beam waist given by [63]

\[ \delta x = \frac{4 \lambda f}{\pi d} \quad (2.23) \]

in which \( f \) is the focal length, and \( d \) is the spot size on the focusing optic. This is, however, tied to the depth of focus, also known as the confocal parameter, \( 2Z_R \) where
$Z_R$ is the Rayleigh range of the beam. The Rayleigh range is defined as the length of the beam from the beam waist (where it is focused) to where the beam has defocused to $\sqrt{2}\delta x$. This relationship is governed by:

$$2Z_R = \frac{\pi \cdot \delta x^2}{2\lambda}$$ (2.24)

So while the axial and transverse resolutions are independent of each other, a relationship does exist limiting the depth at which a feature can be imaged in exchange for higher transverse resolution.
Chapter 3

Apparatus and Experimentation

To investigate the thermal effects described in Section 2.2 and apply them to a model, an in situ imaging modality must be used. The use of OCT in ICI can overcome challenges seen in other in situ imaging modalities. Any strong reflections from the machining beam add incoherently and do not affect the image. This is also true for other light sources that may be present. Design concerns mainly arise from determining the spot size and thus transverse resolution to resolve the feature (here, holes of $<40 \, \mu m$), strong surface reflections due to changing morphology causing saturation, plasma (found when the pulse duration is on the order of the electron-phonon interaction time [64]), and repetition rates that can temporally catch the dynamics of ablation. As well, the use of different imaging wavelengths allows different sets of complimentary data to be taken when exploring silicon and other materials. The experiments involve the investigation of ablation depth for various pulse durations ($<9 \, ps$ vs. $7.2 \, \mu s$ - $44.9 \, \mu s$) and ICI allows this data to be collected without the need to side-polish. As well, ICI can examine the relaxation time scales of liquid silicon and allow a comparison with model results.
3.1 Machining platform

Two machining sources were used for the experiments; a fibre laser with pulse durations ranging from microseconds to quasi-continuous wave (QCW), and a DPSS laser with a pulse duration of 9 ps. The thermal ablation experiments were conducted with the QCW laser (which belonged to the group) while high intensity nonlinear ablation experiments were conducted with the DPSS laser (on loan for 4 weeks). For machining, the polarization was not important to know for the QCW laser, but was required for the DPSS laser as it enabled the use of a quarter wave plate to control power delivered to the sample. Specifications of the sources are expanded upon in Section 3.5.1.

Lenses are used to focus the beams and machining takes place on a stage with the sample set at the focus. The focus itself is determined using an iterative thresholding method to determine the onset of ablation in silicon. This is described in more detail in the next section. The samples themselves are placed on a 3-axis stage allowing quick adjustments to focus and position via motorized control in two directions and manual control via a micrometre in the third. The two motor controlled directions are commanded through digital software with speeds of up to 3 mm/s. This allows the machining of trenches at set speeds as well as blind hole drilling. This main setup includes a nozzle that allows delivery of various assist gases at various pressures. The nozzle was designed by Paul Webster (Ph.D. candidate) with a gas exit hole diameter and barrel length of 0.5 mm. A second machining setup using function generator controlled galvo mirrors and a telecentric lens (LSM03-BB from ThorLabs, 36 mm focal length) allows the movement of the machining beam for the ablation of more complex geometries. Each galvo rotates a mirror and thus moves the machining or imaging beam on the sample. This allows for much faster movement speeds (up to
500 mm/s vs. 3 mm/s for the translation stage) of the machining and/or imaging beam on the sample. The galvo mirrors can also be controlled by software, though this was not used for the experiments shown in this thesis. In contrast, the sample can also be percussion drilled where the machining beam and the sample are both stationary.

3.2 Integrated imaging and machining

The experimental apparatus consisted of the integration of the machining platform and the imaging system via a dichroic mirror. The use of motorized optical flip mounts allows the system to easily change between varying OCT imaging modalities as well as machining methods. Fig. 3.1 shows two different machining setups where a) is designed for percussion drilling of holes (used for majority of experiments) and b) is able to ablate more complex features with the use of galvo mirrors and a telecentric lens. Polarization controllers are used to maintain polarization of the imaging beam and maximize the signal to the detector array while the optical isolator ensures that returning light from the sample does not reenter the source causing damage. Neutral density (ND) filter wheels and focusing lenses in the reference arms attenuate returning light from the reference arm and allows the ability to reduce or remove camera saturation effects. The grating spreads out the returning light in wavelength, which is then focused onto the 1-D camera array. Calibration measurements are conducted before imaging using argon and neon sources, which involves matching pixel numbers to wavelengths from peaks given in National Institute of Standards and Technology (NIST) data for these sources. Without the machining setup, Fig. 3.1 is simply a Michelson interferometer as seen in Fig. 2.9. The dichroic mirror allows transmission at set wavelengths and reflection at others, which enables the machining and imaging
CHAPTER 3. APPARATUS AND EXPERIMENTATION

Figure 3.1: Experimental apparatus for a) percussion drilling and b) drilling of complex feature. Figure used with permission from [65].
beams to be co-axially aligned. This is done by adjustments with two mirror control. While the majority of imaging is done in alignment, the mirrors can also be adjusted to image near but outside the feature as well. To isolate the interferometric term in Eqn. 2.21, the intensities from the sample and reference arms alone are measured a priori and subtracted from the data acquired during imaging. The nozzle present in Fig. 3.1 a) allows for the delivery of an assist gas to aid in the machining process. The imaging beam returning from the reference and sample arms also suffer from a group velocity mismatch, ultimately resulting in the broadening of the PSF. This is known as dispersion mismatch and is corrected by the lens and other optics (i.e. BK-7 glass) in the reference arm.

As the machining and imaging beams are two different wavelengths, this will cause them to focus to different spot sizes as well as different positions in the axial direction. This offset in z can be calculated theoretically and verified with ICI, and the Rayleigh range of the imaging beam is sufficient for the features ablated. To accurately determine the focus of the machining beam (the more important focal distance for ablation), an iterative thresholding method allows ICI to determine a smaller and smaller range where ablation can occur for silicon with decreasing pulse energies. This allows the focal distance to be known and is set to a specific path length delay on the ICI system which is assumed to be accurate barring major realignments. Accurate spot size measurements are made with a camera (PixeLINK PL-A741) and a custom LabVIEW VI made by Paul Webster and Mitchell Anderson (Ph.D. candidate). The spot size determination is another check on the focal distance and path difference between the two focal distances as ICI is able to image the position of the detector array when it is at the beam waist.
3.3 Image processing considerations

Figure 3.2: Gaussian fit (red line) of the imaging spectrum (black line). The imaging spectrum is collected from a linear array and thus is plotted in terms of pixel number. This is converted to wavelength via a determined calibration equation. The peak of the spectrum is 805 nm with a FWHM of approximately 50 nm.

The main imaging program is a LabVIEW VI written by Paul Webster with additions made by the author. The purpose of this program is to view images immediately after ablation to provide feedback and so is streamlined for fast processing. For post-processing, there is no time constraint and more advanced algorithms can be applied.

As seen in Fig. 2.10, each A-line is taken from a FFT of the interferogram in k-space. However, the detector pixels are calibrated to wavelength and thus must be converted into k-space. This is not evenly spaced and thus interpolation is required before an FFT can be taken. Linear interpolation is used by the main program while cubic spline interpolation (added by the author) leads to smaller PSFs in the final image. This comes at a cost of more than 4 times slower processing speed.

Unlike the spectrum used in theory, the spectrum of imaging sources are not Gaussian, leading to PSF broadening. This is improved by fitting a Gaussian to the original spectrum to obtain a multiplication factor to apply to the imaging data pre-FFT. Fig. 3.2 shows the spectrum for the 805 nm imaging system - one of two
imaging centre wavelengths used (see Table 3.2) - with a Gaussian fit. Since the Gaussian fitting is calculated purely from the reference arm signal and involves only a multiplication factor to the final signal, it is not a time intensive process and is used during experimentation as well as post-processing. During post-processing, both Gaussian fitting and the aforementioned cubic spline fitting is applied using Matlab code written by this author with contributions from Ben Leung (former M.A.Sc. candidate). The post-processing also includes setting the final dynamic range of the image discussed briefly in the next section.

3.4 Imaging modes

When the imaging beam is stationary as in the case with percussion drilling, depth information is collected as a function of time. These are known as motion mode, or M-mode images. Fig. 3.3 shows a simulated M-mode of one interface with the time and depth as the x- and y-axes respectively. The bar to the right of the figure is the reflectivity scale. At the start, the interface is unchanged and held at position of 200 \( \mu m \) corresponding to the optical path length difference from zero delay. After a certain time, the interface moves away from zero delay, which is seen in the image. Time in a real image would be determined by the repetition rate of the imaging beam and the number of A-lines. Each A-line gives depth information at a specific time and thus every M-mode is composed of a set of A-lines. The brightness of an interface represents its reflectivity and has units of dB and is a logarithmic scale referenced to the sensitivity of the system. This sensitivity is determined from the lowest detectable signal from a mirror after passing through an ND filter.

Typical sensitivities for SD-OCT systems are \( >80 \text{ dB} \) \cite{66} and images will often span a specific dynamic range (\( e.g. -20 \text{ to } -60 \text{ dB} \) rather a full range from 0 to -80 dB)
Figure 3.3: Simulated M-mode image of a single interface that is at first static and then moves away from zero path delay.

to highlight features of interest due to limitations of dynamic range placed by the camera and print media in accurately reflecting the full sensitivity. The sensitivity range spans from 100% reflection from the sample arm (i.e. all the imaging light returns to the camera) to the noise floor, represented by 0 dB and some negative value, respectively. Thus the reflectivity in units of dB is defined as:

$$Reflectivity = 10 \log \frac{I_{Sam}}{I_{SamPerfect}}$$  \hspace{1cm} (3.1)

where $I_{Sam} = E_{Sam}^2$, $I_{SamPerfect} = E_{SamPerfect}^2$, and $E_{SamPerfect}$ is perfect reflection from the sample arm. It can be seen that this can be simplified to:

$$Reflectivity = 20 \log \frac{E_{Sam}}{E_{SamPerfect}}$$  \hspace{1cm} (3.2)

$$= 20 \log \frac{I_{Cross}}{I_{CrossPerfect}}$$  \hspace{1cm} (3.3)

where $I_{Cross} = E_{Sam} \cdot E_{Ref}$, and $I_{CrossPerfect} = E_{SamPerfect} \cdot E_{Ref}$. The m-mode images
show intensity and thus, an image displaying the entire sensitivity range of a system with a noise floor 8 orders of magnitude weaker than perfect reflection would have a reflectivity ranging from 0 to -80 dB.

The galvo mirrors shown in Fig. 3.1 b) can also be used to scan the imaging beam. In this case the image generated is closer to traditional OCT with two spatial dimensions, known as B-modes (where B stands for brightness). By scanning in a third direction, these images can be combined to create 3-D renderings of the features. Fig. 3.4 shows a set of holes drilled and imaged by evenly spaced OCT B-modes and compiled into a 3-D rendering. The imaging beam is incident from the top of the image and the unevenness of the top surface is due to recast. Above the top surface (at 100 µm) is air, and below (except where the holes are present) is stainless steel that the imaging beam cannot penetrate. The bottom of the holes are at a depth of 320 ± 10 µm. This figure was taken using the 1320 nm imaging system - the second of two imaging systems described in Section 3.5.2 (see Table 3.2). The axial and transverse resolutions are determined by Eqns. 2.22 and 2.23 respectively.

Outside of OCT, optical microscopy alongside side-polishing is also used to provide
information about the quality of the cut on the surface and inside the feature.

### 3.5 Light sources

#### 3.5.1 Machining

Most of the work with silicon was conducted with a 100 W fibre laser centred at 1070 nm with pulse durations ranging from 7.2 \(\mu\)s to QCW. The variability in pulse length and power allowed exploration of a wide parameter space. The time regimes are long and all ablation pathways are thermally based. In contrast, a short pulse system (<9 ps) was also used to compare high intensity nonlinear ablation as well as processing of other materials. This system was on loan for four weeks from Altos Photonics. One of the major advantages of the fibre system is the ease of delivery into the machining platform as well as exposure control. A transistor-transistor logic (TTL signal) from a signal generator was sufficient for controlling the number of pulses delivered to the sample to within 1 pulse. The longer warm up time (to obtain sufficient pulse energy) for the picosecond system required a custom-made exposure control in the form of a single-lens reflex (SLR) camera. Once the laser had finished its warm-up cycle, a TTL command was sent to the camera for a specific exposure duration. The unfocused nature of the beam (FWHM ~2 mm) where the SLR camera was positioned limited the damage. As mentioned previously, pulse energies were scaled using a quarter-wave plate for the short pulse system and the QCW laser allowed the value to be digitally set. While the short pulse system allowed for cutting without thermal effects, it came at the expense of an order of magnitude increase in cost (~$170,000 for the Ekspla vs. ~$10,000 for the IPG). Another advantage of the fibre laser is a much higher \(M^2\) factor (1.05 vs. ~1.5), which describes the quality of the beam by its resemblance to a Gaussian beam profile where a \(M^2\) of one is a
perfect Gaussian. Finally, fibre delivery of the QCW laser makes it far more robust than the free-space short pulse laser and as such, is easier to integrate into industry. Table 3.1 summarizes the characteristics of the machining sources.

Table 3.1: Machining source data. Adapted from [60].

<table>
<thead>
<tr>
<th>Light Source</th>
<th>QCW Yb-fibre laser</th>
<th>Diode pumped solid-state laser</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center wavelength, λ₀ (nm)</td>
<td>1070</td>
<td>1064</td>
</tr>
<tr>
<td>Pulse Width</td>
<td>6.7 μs - QCW</td>
<td>9 ps</td>
</tr>
<tr>
<td>Maximum Average Power at Sample (W)</td>
<td>100</td>
<td>6.9</td>
</tr>
<tr>
<td>Repetition Rate (kHz)</td>
<td>CW - 50</td>
<td>50-100</td>
</tr>
</tbody>
</table>

3.5.2 Imaging

Two different centre wavelengths were used for OCT imaging at 805 nm and 1320 nm. The longer wavelength had the interesting effect of imaging through the silicon and thus allowing the visualization of the bottom interface. For other work done by the group, this wavelength also leads to better penetration when imaging in biological samples. However, standard silicon detectors cannot be used and the InGaAs array has a limited A-line rate compared with the silicon detector for 805 nm as well as a lower axial resolution (given the same spectral width as seen in Eqn. 2.22).

For the 805 nm source, a femtosecond Ti:AlO₂ oscillator is used. This is unconventional for OCT imaging as the system is far more expensive than other available sources at this wavelength. What is interesting is the broadening of the spectrum that occurs after being coupled into fibre. This was set up by Paul Webster and the exact nature of the broadening is not well understand and is a subject of ongoing research [67]. Table 3.2 gives a summary of the imaging sources.

To synchronize the imaging and machining beams such that an A-line image is
Table 3.2: Imaging system data. Adapted from [60].

<table>
<thead>
<tr>
<th>Light Source</th>
<th>Ti:AlO$_2$ Oscillator</th>
<th>Coupled SLD Pair</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coherent Mira 900</td>
<td>InPhenix IPSDM-1322</td>
</tr>
<tr>
<td>Detector</td>
<td>Silicon Detector</td>
<td>InGaAs Detector</td>
</tr>
<tr>
<td>Basler Sprint spL4096-140km</td>
<td>805</td>
<td>1320</td>
</tr>
<tr>
<td>Goodrich SU512LDV-1.7RT-0500/LX</td>
<td>14</td>
<td>98</td>
</tr>
</tbody>
</table>

Center wavelength, $\lambda_0$ (nm) 805 1320
Axial resolution, $\delta z$ ($\mu$m) 5 14
Sensitivity (dB) 103 98
Maximum A-line Rate (kHz) 312 24
Temporal Resolution ($\mu$s) 1.5 8

taken during the machining pulse, both systems are triggered by a dual output function generator. The electronic delay between trigger and machining pulse activation, and trigger and camera acquisition can be measured by a photodiode and an output signal from the imaging camera respectively; this delay is then accounted for in the function generator. The imaging A-line acquisition rate can then be set to a multiple of the machining repetition rate. This maintains when A-lines are acquired during and after a pulse, with respect to the start of the pulse.
Chapter 4

Results for Silicon

This chapter will serve to show the dynamics of thermal ablation in silicon as well the quality of the cuts that can be achieved. M-mode images shed light on the relaxation times for the sample after the pulse interaction as well as the rates at which ablation occurs. Modeling also shows that pulse-to-pulse buildup effects seen by other groups is not a factor. A comparison is also made between thermal and nonlinear ablation.

4.1 Thermal ablation with assist gas

As an initial proof of concept, it was important to show that not only is cutting possible, but quality cutting can occur without detrimental heat effects that would restrict features to limited use for industrial applications. One commonly used control parameter found to improve quality and the material removal rate is assist gas [27, 68, 69]. Fig. 4.1 shows M-modes taken of ablation in stainless steel showing the difference in dynamics when no gas, nitrogen, and oxygen are used. The assist gases were delivered co-axially with the machining beam with a surrounding environment at atmospheric conditions. While all three show signs of depth saturation, it is clear
Figure 4.1: Comparison of assist gas effects in 304 stainless steel where a) uses no gases, b) uses $N_2$ at 6.8 bar and c) uses $O_2$ at 6.8 bar. Adapted from [27].

that the oxygen assisted reached this depth much faster. In all three images, there is another interface at a much shallower depth. This is due to the imaging beam having a larger transverse resolution (35 $\mu$m) than the bottom of the hole and so sidewall features can be seen. This can be an advantage as some 2-D information is inadvertently obtained. For these holes, 100 $\mu$s pulses with an energy of 10 mJ at a rate of 4 kHz were used for ablation (2500 pulses total). For this and all subsequent M-modes, the machining and imaging beams are incident from the top of the image unless otherwise stated. This was imaged using the 1320 nm system with an A-line
CHAPTER 4. RESULTS FOR SILICON

Figure 4.2: Ablation of silicon a) without assist gas and b) with N\textsubscript{2} assist gas at a pressure of 6.2 bar. TI stands for top interface, SW for sidewalls, and EM for end of machining laser exposure. The green lines are intended as a guide to the ablation front. The imaging rate is 300 kHz while the machining rate is 5 kHz giving 2 A-lines during and 58 A-lines after the pulse interaction.

For silicon, the longer pulse durations used for stainless steel caused severe microcracking along the rest of the sample and shorter pulses were required. Fig. 4.2 shows the ablation in silicon a) with, and b) without nitrogen assist gas. The silicon in the experiments was n-doped, crystallographic orientation <111>, with a thickness of 275 \(\mu\text{m}\). End of machining laser exposure (EM) occurs after 24 pulses and the sample is seen to remain static after this time. The machining pulses were of 7.2 \(\mu\text{s}\) duration at a repetition rate of 5 kHz with a pulse energy of 560 \(\pm 10\) \(\mu\text{J}\). The 805 nm imaging system was used at an A-line rate of 300 kHz giving an imaging ratio of 60 frames to one pulse, where two frames are imaged on the pulse itself.
For some of the A-lines, saturation effects can be seen with the bright interface below the ablation front (0-0.5 ms in Fig. 4.2 a) and just after 1 ms in Fig. 4.2 b). While ICI is able to image multiple layers in some cases, 805 nm does not transmit well through silicon and the saturation effect is verified when analyzing the raw pixel data (which showed many pixels with values corresponding to maximum brightness). Also, the high reflectivity of the ablation front at certain points leads to the effect of acting as another reference arm. This resulted in what appears to be layers above the top of the silicon and is an imaging artefact. There are several features of interest to note from Fig. 4.2: the saturation in depth of ablation, the increase in sidewalls (SW) without assist gas compared to with assist gas, and the time scales at which liquid silicon solidifies. These are discussed below.

First, the ablation front saturates at a depth of 180 $\mu$m and 200 $\mu$m with and without assist gas respectively. This is highlighted in Fig. 4.2 by the two green lines with very different slopes, the first showing where material removal is fastest, the second where material removal has almost completely stopped. This is a curious effect as both depths are still within the Rayleigh range of the machining beam ($\sim$300 $\mu$m in air). The effect is one that can be seen through in situ imaging after one hole, whereas ex situ methods would require several along with time consuming side-polishing processing. One possible explanation for the depth saturation is an inability of the material to escape the hole due to the high aspect ratio. This result has been seen by other groups and is a matter for future research [70-72].

Second, the machining spot size was measured to be 20 $\mu$m FWHM, resulting in features of $14 \pm 1$ $\mu$m in diameter as seen in Fig. 4.3. This gives another possible explanation of the saturation effect as beam clipping due to the machining beam being larger than the hole diameter. The smaller hole size compared to the beam is also surprising as it suggests an optical thresholding effect. Given the imaging beam
Figure 4.3: Optical microscopy images of holes machined in silicon a) without assist gas and b) with N\textsubscript{2} assist gas at a pressure of 6.2 bar. These are the same holes shown in the M-mode images of Fig. 4.2. The images were taken after both features were cleaned with an ultrasound bath [26].

size of 23 \( \mu \text{m} \), information is obtained about the surface or top interface (TI) of the material as well as the sidewalls. Without assist gas, the ablation front appears to be more stochastic as well as having a thicker sidewall suggesting a narrower hole, confirmed in Fig. 4.3. Interestingly, the final depth of the ablation is shallower when nitrogen assist gas is present suggesting that the gas has a cooling effect on the sample reducing the amount and rate of vaporization. A much more significant effect is seen from top view optical microscopy images (Fig. 4.3) where the outside surface of the holes is significantly cleaner when nitrogen assist gas is present. The amount of debris present without assist gas may account for some of the extra features in the corresponding M-mode. As well, in both cases the holes have a desirable high aspect ratio of approximately 10. Also, using Eqn. 2.17, the experimental machining spot size of 20 \( \mu \text{m} \) gives a top hat equivalent area of 160 \( \mu \text{m}^2 \). With a hole diameter of 14 \( \pm \) 1 \( \mu \text{m} \) seen in Fig. 4.3 b), the area of the hole is 150 \( \pm \) 10 \( \mu \text{m}^2 \), which is a good match to the top hat equivalent area.

Finally, in Fig. 4.2, it can be seen that only the first two or three A-lines after
Figure 4.4: Closeup of Fig. 4.2 b) showing the interaction of the sample for machining pulse. The red line indicates when the machining pulse is active on the sample. Each vertical line of pixels represents one A-line.

the machining pulse changes before settling to an equilibrium depth. This gives rough time scales of how fast the silicon cools and re-solidifies. Fig. 4.4 shows a closeup view of Fig. 4.2 b) after the second machining pulse, suggesting that the layer becomes static after approximately 3-4 A-lines (10-13 µs). Modeling results present in Section 4.3 will show that the layer will have cooled to room temperature before the next pulse, implying a faster repetition rate is possible without any change to the cut quality. Unlike what has been published by [73, 74] who describe a pulse-to-pulse thermal build-up effect (termed multi-pulse enhancement effect), this is not the case for the experiments conducted with a 5 kHz machining laser repetition rate.

4.2 Pulse duration effects

The experiment that was compared to modeling was the ablation of silicon with no assist gas, constant pulse energy and repetition rate (thus constant average power), but
Figure 4.5: Shapes of pulses used in the experiment and modeling simulation. A varying pulse duration. Even though the amount of energy in the pulse remained unchanged, the shortening of the pulse duration yields higher intensities. The thermal nature of the process also suggests that less thermal diffusion would occur for the shorter durations, possibly leading to more energy contributing to material removal. The average power for each pulse duration was verified to be the same with a calibrated thermal detector. Fig. 4.5 shows some of the pulses used in the next set of results.

For all these pulses, the pulse energy and repetition were kept at a constant $560 \pm 10 \mu J$, and $5 \text{ kHz}$, respectively while the pulse durations were varied, resulting in different intensities. All the pulses have a spike at the beginning with a width ranging from $0.35 \mu s$ FWHM for the shortest pulse and $1.3 \mu s$ FWHM for pulses longer than $10 \mu s$. This is a characteristic of the QCW fibre laser. Since the majority of energy is contained in the tail of the pulse, it is inappropriate to use a FWHM definition for pulse length. Instead, the pulse length is defined as the tip to tail duration when the instantaneous power is above the noise floor of the detector at
Figure 4.6: M-mode images of ablation in silicon for a pulse duration of a) 11.2 $\mu$s and b) 32.7 $\mu$s. No assist gas is used. The inset image in a) shows the shape of the hole and reason for the double interface at the bottom.

6 W of power.

Fig. 4.6 shows the M-mode images of ablation in silicon with machining pulse durations of a) 11.2 $\mu$s, and b) 32.7 $\mu$s, and a total of 24 pulses. Green lines are added to highlight the ablation front and it can be seen that depth saturation occurs for the shorter pulse duration but not the longer. Of note is the double interface at the bottom of Fig. 4.6 a), which represents the bottom of the silicon and an outcropping just above. This hole shape is shown in the inset image in Fig. 4.6 a). For both images, saturation effects are seen and is prominent for Fig. 4.6 b); interfaces $\sim$150 $\mu$m below the ablation front in Fig. 4.6 b) are purely due to saturation and are thus imaging artefacts. From Fig. 4.6, it can be seen that though the pulse energy is the same for both cases, the change in pulse duration yielded a much fast ablation rate (before depth saturation) and more material removal for the shorter pulse.
Figure 4.7: Per pulse ablation rate as a function of a) pulse duration and b) pulse intensity. Pulse energy is kept constant for all data points at 560 ± 10 µJ.

Fig. 4.7 shows the ablation depth per pulse (before depth saturation) as a function of a) pulse duration and b) intensity. The plots show that the ablation rate dramatically increases with shorter pulse durations and thus higher intensities, where pulse intensity is defined as pulse energy/(pulse length × spot size of focused beam).

By changing the pulse duration, an inadvertent effect is a change in the pulse shape as the shorter pulses have a higher spike. Thus, there are two effects which may cause the increase in ablation rate. These two effects cannot be experimentally isolated and so a numerical model is used to determine which is dominant.

### 4.3 Modeling results

To obtain the most accurate comparison to the data, modeling was done for the case of no assist gas and with the intensity entering into the interaction volume having the same distribution as pulse shapes from the experiment. The model simulates the heating and cooling of the interaction volume as it interacts with a defined pulse (in
Figure 4.8: Simulation of temperature profile in interaction volume for 7.2 µs pulse with constant absorption corresponding to room temperature. The red line on the time axis indicates when the machining pulse is on. As the absorption depth is much longer than the thickness of the sample, any rise in temperature is more evenly distributed throughout the depth. Only the first 38 µm are shown for clarity of image.

This case, the pulses of various duration used in the experiment, see Fig. 4.5). The model outputs the temperature distribution throughout the volume for every time step of the simulation (as seen in Fig. 4.8 and 4.9) as well as the amount of material removed.

Fig. 4.8 shows the temperature profile for interaction with the 7.2 µs duration pulse if the absorption depth remains constant at 1 mm, corresponding to room temperature. The volume only rises to a temperature of 629 K, far below the melting and vaporization temperatures of 1687 K and 3538 K for silicon. This shows that thermal ablation at the settings used cannot occur without the nonlinear change in absorption.

Other research groups show an analytical model of silicon ablation with nanosecond pulses at a wavelength of 1070 nm and make the simplification that the absorption coefficient is constant at a silicon temperature of 550 K [12, 75]. The appropriateness
Figure 4.9: Simulation of temperature profile in interaction volume for 7.2 $\mu$s pulse with temperature dependent absorption. The red line on the time axis indicates when the machining pulse is on. Unlike in Fig. 4.8, the absorption depth greatly shrinks as the volume heats up and surface layers become much hotter than below. For the lower depths, the main cause of rising temperature is now heat diffusion from above as opposed to interaction with the pulse. Only the first 38 $\mu$m are shown for clarity of image.

of this simplification can be evaluated by applying it to the model presented here. It was found that only a minimal removal rate is achieved for the 7.2 $\mu$s pulse duration (2.1 $\mu$m/pulse), and temperatures for ablation could not be reached for all other pulses.

It is interesting to note that in Fig. 4.8, the temperature at a given time is uniform throughout the depths, which is unlike Fig. 4.9 where the temperature drops off rapidly as one goes deeper into the sample. This is because temperature rise for all slices in Fig. 4.8 is directly from the pulse as the absorption depth is much longer ($\sim$1 mm) than the thickness of the sample ($\sim$275 $\mu$m) and held constant at room temperature. When nonlinear absorption is included in the model, the absorption depths shorten to the same order as the slice thickness (0.6 $\mu$m absorption depth at
1687 K vs. 0.125-0.28 \( \mu \text{m} \) slice thickness) resulting in the pulse energy being deposited only in the top layers. Thus the topmost layers are extremely hot followed by a steep gradient to the lower depths. In this case, the lower depths are heated indirectly by heat diffusion from the top layers. The steep gradient is seen in Fig. 4.9. As well, the temperatures in Fig. 4.9 reached a quasi-equilibrium state while the temperatures continue to rise in Fig. 4.8 when the machining pulse is on. This is because the energy entering the interaction volume during the quasi-equilibrium state went into maintaining the temperature gradient and material removal instead of changing the temperature gradient. This is expanded upon later.

These results show that indeed there is a cascaded absorption effect where the early parts of the pulse slowly begins to heat up the sample so that more energy from the later parts can be absorbed.

Once a layer has vaporized in the simulation, it is removed from the model. Fig. 4.10 shows the temperature profile slices at time 2 and 6 \( \mu \text{s} \) of Fig. 4.9. The curves do not start at a depth of zero signifying that those earlier depths have been ablated. This can be seen as the curvature at the top of Fig. 4.9. From Fig. 4.9, information can be extracted about the temperature gradient in the volume, and the depth removed per pulse.

Fig. 4.11 shows the temperature profile slices at time 2, 6, and 30 \( \mu \text{s} \) for the 38.5 \( \mu \text{s} \) pulse. The spike is able to heat up the volume to vaporization, but this temperature gradient is unable to be maintained once the pulse flattens and thus ablation only occurs during the spike. This is expanded upon later in the section.

Fig. 4.12 shows the experimental ablation depth per pulse along with predicted values from the model. The ablation rates are calculated from the steep slope at the start of the ablation as seen in Fig. 4.2 and does not include the saturated cutting region. As the data points used corresponded to experimental pulse shapes, the
Figure 4.10: Temperature profiles of the interaction volume 7.2 µs pulse at 2 (black curve) and 6 (red curve) µs corresponding to the same time on Fig. 4.9. For both curves, vaporized earlier depths are removed from the calculation.

Figure 4.11: Temperature profiles of the interaction volume for 38.5 µs pulse. Of the three times, 6 µs shows the volume at its hottest after initial heating from the spike. At 30 µs when the pulse has flattened out, the surface temperature is below what is required for vaporization.
CHAPTER 4. RESULTS FOR SILICON

Figure 4.12: Experimental and simulated ablation depths per pulse. Pulse energy is kept constant for all data points at 560 ± 10 µJ.

Simulated results are discrete points and not a continuous curve. Using only \( L_{\text{Radial}} \) as a fitting parameter (discussed in Section 2.3.2) a fit to the data with a reduced chi square value of 4.3 is achieved. The modeling fit suggests a higher ablation rate than a majority of experimental results. This is due to the fact that going to a longer \( L_{\text{Radial}} \) would give the two lowest intensity pulses (with relatively small uncertainties, thus having a greater weighting in the reduced chi square calculation) too low an ablation rate. Other simulations with the model show that no liquid silicon is present in the interaction volume approximately 10 µs after the pulse, in agreement with Fig. 4.4, which shows an unchanging interface after 3-4 A-lines (10-13 µs). Also, if ablation is assumed to be a linear optical process (simplest model), ablation rate would be dependent entirely on and scale linearly with pulse energy; there would be no difference in ablation rate for the same pulse energy. This is not seen in Fig. 4.12 which indicates a nonlinear effect.

Fig. 4.13 shows the reduced chi squared value with varying \( L_{\text{Radial}} \). With too short
Figure 4.13: Reduced chi squared values for varying $L_{Radial}$. The dramatic increase at very long and short distances show that there is an optimal value for fitting purposes and agrees with expected trends.

of a distance, radial diffusion completely dominates heat loss and ablation cannot occur. When the distance is set too long, heat diffusion out of the volume slows down greatly and leads to a gross overestimation of material removal. This behaves as expected and $L_{Radial}$ corresponding to the minimum chi squared value is used in the modeling calculations.

Given good agreement between the trends seen in the model and experiment, the model can be used to simulate the effects of changing various pulse parameters, specifically, pulse duration and height of the initial spike independent of each other. Fig. 4.14 shows the ablation depth per pulse for temporal top hat pulses with durations corresponding to the experimental pulses. The figure follows the general trend seen in Fig. 4.12, suggesting that the dominant parameter affecting ablation rate is the peak intensity. However, top hat pulses appear to be unable to initiate ablation for the shortest two pulse durations. Also, for pulses where ablation does occur, the ablation
Figure 4.14: Ablation depth per pulse for simulated top hat pulses with same pulse energy. Inset image shows the pulse shape.

rate is higher than the simulated ablation rates for the experimental pulses.

Fig. 4.14 shows that the cascaded absorption process is nonlinear but not in a standard multiphoton way that would achieve better ablation rates with higher intensities. The curvature suggests a more complicated relationship than a simple $I^2$ or $I^3$ process and thus leads to an uncertainty of the ideal pulse shape. An investigation of the initial spike provides additional information on the effects of pulse shaping.

Fig. 4.15 examines the effect on ablation rate of varying the height of the initial spike for the a) 38.5 $\mu$s and b) 32.7 $\mu$s pulse (4.63 MW/cm$^2$ and 5.62 MW/cm$^2$ respectively) while maintaining the same pulse energy. The width of the spike was set as the duration between tip and when the pulse flattens for the experimental pulses (3 $\mu$s). Fig. 4.16 shows the same data as Fig. 4.15 with the x-axis now representing the proportion of energy in the spike; the dependent variable is now linear with spike energy. Fig. 4.16 more clearly shows the effects that are linear with intensity and an extrapolation of the data suggests an ablation rate of approximately 19 $\mu$m/pulse.
Figure 4.15: Ablation depth per pulse for varying spike heights for pulse durations of a) 38.5 µs and b) 32.7 µs with same total pulse energy. The inset image in a) shows the pulse shape used in the simulation for both a) and b). The error bar on the fifth point in both figures show the simulation spatial step and thus discretization uncertainty of the simulation. For both plots, pulse energy is kept constant at 560 ± 10 µJ.

when 100% of the energy is in the spike.

In Fig. 4.15 a), ablation begins when the spike to flat portion ratio (SFR) is a factor of 4, giving an ablation rate that matches the one seen in Fig. 4.12; this SFR is approximately the same as the experimental pulses. Fig. 4.15 b) shows that, initially, increasing the proportion of energy in the spike is detrimental to the ablation rate. Thus, for the SFRs of the experimental pulses, the spike aids in initiating ablation for the longer duration pulses but negatively affects the ablation rate for the shorter duration pulses.

For the shorter duration pulses with higher peak intensities, a set amount of energy is used to maintain the temperature gradient, which is now at a quasi-equilibrium once vaporization of the material occurs, and any energy above this determines the material removal. For the longer duration pulses with lower peak intensities, only the initial spike has enough energy to remove a few layers before the energy drops
Figure 4.16: Ablation depth per pulse for varying spike heights with same total pulse energy as a function of energy in the spike for pulse durations of a) 38.5 µs and b) 32.7 µs. The error bar on the fifth point in both figures show the step size and thus discretization error of the simulation. For both plots, pulse energy is kept constant at 560 ± 10 µJ.

This figure shows the ablation depth per pulse for varying spike heights with the same total pulse energy as a function of energy in the spike for pulse durations of a) 38.5 µs and b) 32.7 µs. The error bars on the fifth point in both figures indicate the step size and thus the discretization error of the simulation. For both plots, the pulse energy is kept constant at 560 ± 10 µJ.

The flat portion of the curves indicates that the quasi-equilibrium surface temperature (at 30 µs) is below the vaporization temperature and thus ablation only occurs during the spike. As a result, the factor that most affects ablation rate for thermal ablation is not instantaneous or average power, but pulse intensity (determined from pulse shape and duration) once quasi-equilibrium is reached during vaporization. This is evident when comparing the amount of heat diffused out of the volume for the 16.3 µs pulse (11.1 MW/cm²) with the 32.7 µs pulse (5.52 MW/cm²). At the end of pulse, approximately 52 µJ of energy (8.8% of the total pulse energy) diffused from the longer duration than the shorter duration pulse, which is the energy required to maintain the temperature gradient for the longer time. If the 52 µJ went into material removal instead, a rough calculation yields an additional 8.2 µm of material removal. This is approximately the difference in ablation rates both experimentally and simulated between the 16.3 µs
pulse (11 ± 1 µm/pulse and 13.4 ± 0.1 µm/pulse respectively) and the 32.7 µs pulse (2.9 ± 0.5 µm/pulse and 4.5 ± 0.1 µm/pulse respectively); a difference of 8 ± 1 µm/pulse and 9.0 ± 0.3 µm/pulse respectively.

The drop in ablation rate from a SFR of 4 to 5 in Fig. 4.15 b) is where the flat portion can no longer maintain the quasi-equilibrium and ablation is now entirely due to the spike. Thus, after a SFR of 4 the flat portion no longer contributes to material removal and the increase in ablation rate is purely due to the increase in energy in the spike. Fig. 4.11 confirms this as well with the quasi-equilibrium surface temperature (at 30 µs) below the vaporization temperature showing that ablation only occurs during the spike. The rise in ablation rate from an SFR of 2 to 3 in Fig. 4.15 b) represents the point where the spike is now able to initiate cutting, but the flat portion still has sufficient energy to maintain its material removal rate. If a trend is extrapolated backwards for SFRs of 5 - 10, it can be seen that it crosses the x-axis at an SFR of 2 and shows cutting beginning at an SFR of 3. Therefore, Fig. 4.15 b) shows two effects, the spike and the flat portion both contribute to cutting. As energy is redistributed into the spike, the overall ablation rate decreases until the spike becomes the dominant material removal mechanism and the flat portion no longer contributes.

The simulated results show that there is a competing effect between the percent of energy in the spike and the total pulse duration and an optimum pulse shape that maximizes the ablation rate exists. Overall, ablation rate increases with higher intensity (shorter duration) pulses with constant pulse energy. However, pulse duration is limited in real sources, which may have more control over pulse shapes. The work here shows that an ideal pulse shape is highly dependent on the energy and duration of the pulse. An initial spike can both aid and hinder ablation rate and should be tailored accordingly.
4.4 High intensity nonlinear ablation

High intensity nonlinear ablation (shortened to nonlinear ablation) is a different regime of ablation where the material removal is not dominated by melt or vaporization, but instead electronic effects from the high intensity beam due to a very short pulse.

Fig. 4.17 shows an M-mode measurement of nonlinear ablation in silicon with the Ekspla PL10100 giving pulse durations of 9 ps, a pulse energy of 138 $\mu$J, a repetition rate of 50 kHz, and focused to an 18 $\mu$m FWHM spot. The 805 nm imaging setup is used, again giving an A-line rate of 300 kHz and thus a ratio of 6 A-lines per machining pulse. What appears to be a single pixel is in actuality a combination of 6 A-lines. Thus, the M-mode shows no relaxation dynamics, which is in agreement with the absence of any melt as expected from nonlinear ablation. Fig. 4.17 is scaled to show the same amount of energy delivered (32 pulses) as the first 8 pulses in Fig. 4.2 b) (the regime before depth saturation). No assist gas is used. In comparing the same energy delivered, it can be seen that thermal ablation with the 7.2 $\mu$s pulse, while more stochastic, achieves a higher per energy ablation rate (0.04 $\mu$m/$\mu$J vs. 0.01 $\mu$m/$\mu$J) and thus is more efficient for material removal along the z-direction. Note that the thermal ablation laser costs an order of magnitude less than the high intensity laser (see Section 3.5.1).

Fig. 4.17 and Fig. 4.18 shows a hole cut with a fluence of 44 J/cm$^2$, which is in the industrially relevant regime described in Section 2.4.

Fig. 4.18 shows a top down optical microscopy image of the hole from Fig. 4.17. The entire hole was exposed to the machining beam for 4 ms. While the ablation process does not require thermal melt and vaporization, the edges of the hole continue to show damage to the top surface. Though the high intensity beam ablates via CE
(see Section 2.4), this occurs only in the first hundreds of femtoseconds [76, 77]; the layers below where CE takes place are rapidly brought to above critical temperatures and easily ionized occurring on the order of tens of picoseconds [78]. The high energy plasma is now free to damage the surface of the material. This is known as plasma etching and is a problem found with some high intensity ablation (dependent on material and laser parameters).

The highly deterministic nature of nonlinear ablation allows continuous imaging of the back interface of silicon using the 1320 nm system. Fig. 4.19 shows what appears to be the back interface rising to meet the ablation front as machining progresses. The nature of OCT measures optical path length difference and the higher index of refraction (3.5 for a wavelength of 1320 nm [34]) in silicon makes the distance appear longer than it is (∼950 μm thickness before any material is removed). Thus, the light travels through less of the higher index medium as silicon is removed resulting
CHAPTER 4. RESULTS FOR SILICON

Figure 4.18: Optical microscopy image of nonlinearly ablated hole in silicon. No post processing (i.e. ultrasound bath) is done to the sample.

Figure 4.19: Ultrafast ablation of silicon imaged by the 1320 nm OCT system [79].

in the rising back interface. The two other interfaces seen in the figure are results of imaging through sidewalls. The two fronts meet 2.7 s after the start of ablation showing perforation at 275 µm, matching the wafer thickness.

This is one of the biggest advantages of ICI as not only can it guide cutting to a specific depth, but given the right material and wavelength ICI can guide cutting to a specific distance from the other side of the material without the need to obtain a priori information about the thickness of the sample. ICI is a potential solution to certain applications (e.g. surgery) where a priori information cannot be obtained.
Chapter 5

Ablation of Other Materials and Feedback Control

High intensity ablation of other materials was explored and compared with other fabrication techniques in the literature to determine aspects where improvements can be made in the laser micromachining process. Specifically, lead zirconate titanate (PZT) and PMMA were investigated with the high intensity machining platform for blind hole cutting as well as more complex shapes. These materials are common in ultrasound devices and microfluidics, respectively. Thermal effects such as HAZs are not desired in either material and thus high intensity nonlinear ablation is explored. Studies here show only a preliminary investigation due to the time constraint of the machining laser (on loan for 4 weeks). As well, the nature of ICI makes it an ideal in situ imaging method to provide depth control and the results of manual feedback are presented.
5.1 Fine grain PZT

Figure 5.1: M-mode of nonlinear ablation of PZT using the 805 nm imaging system. The machining pulses terminate after 10 ms. The ablation rate rate is 0.18 $\mu$m/pulse.

Parameters were first optimized for blind hole drilling to determine minimum plasma etching by varying average power. Optical microscopy allowed for an estimate on the quality of the cut. Fig. 5.1 shows a clean and deterministic ablation process in PZT. The image is taken with the 805 nm system at an A-line rate of 300 kHz with a machining rate of 50 kHz. The pulse energy is 3.43 $\mu$J and terminated after 500 pulses (10 ms) The ablation front is very linear and suggests that a target depth can be set by changing the number of machining pulses alone. Also, the PZT appears to transmit a portion of the infrared imaging beam and once again the effect of the rising bottom interface is seen. The sample is perforated after 84 $\mu$m, though the exit hole is smaller than the entry hole giving a reflection at the bottom (corroborated with optical microscopy). The strong reflection from the top surface indicates minimum change due to melt or debris as well as a feature size that is much smaller than the transverse resolution of the imaging beam.
Figure 5.2: Top view of optical microscopy image of nonlinearly ablated hole in PZT. No post processing (i.e. ultrasound bath) is done to the sample.

Fig. 5.2 is an optical microscopy image of the hole from Fig. 5.1. There is discoloration on the surface indicative of a thermal effect but appears limited. The FWHM of the machining beam is 18 µm while the hole is only 12 µm in diameter, suggesting a thresholding effect where only the most intense part of the pulse is able to achieve ablation. This gives an aspect ratio of 7 and yields a volumetric removal rate of roughly 1 µm³/µs. As suggested by Fig. 5.1, Fig. 5.2 confirms that there is minimal change to the surrounding top surface.

With these parameters showing positive results, trenches were next cut using a motorized translation stage to more closely match the features found in high frequency linear array transducers. Lukacs et. al [31] demonstrates an array created using laser micromachining with a wavelength in the ultraviolet. The array consists of trenches 8 µm across, 3 mm long, 80 µm deep, separated by 37 µm with a cutting speed of 1 mm/min. The 64 element array presented took a total of 192 min to create while an extrapolation for the volumetric ablation rate demonstrated here (1 µm³/µs for blind
Figure 5.3: Two sets of four trenches cut in PZT at 3 mm/s (left set) and 2 mm/s (right set).

hole cutting) shows the same array could potentially be created in 4 min. Fig. 5.3 shows two sets of 4 trenches cut at various speeds on top of gold plated PZT. The gold top surface had no effect on the cutting process.

To create trenches, the beam was scanned across the surface by moving the sample with the motorized stage. The scan speed was set to 3 mm/s and 2 mm/s for the set of trenches to the left and right respectively. The pulse energy was 5.3 µJ at a 50 kHz rate and one pass per trench. Various pulse energies and scan speeds were tested and the two best results are shown here. There is no noticeable difference between the two sets on the surface and no thermal damage to the surroundings, which was seen when multiple passes were made. A rough estimate of trench depth via optical microscopy did show the slower cut trenches having a higher variability in trench depth than the faster cut trenches. Both set of trenches had widths of 15 µm, which is non-ideal compared with the 8 µm width presented by Lukacs et al. [31]. However, the two orders of magnitude in improvement in processing speed and a large unexplored parameter space suggests that optimization can lead to far more efficient
methods of production. It was found that higher pulse energies led to damage to the surrounding material and thus more than 98% of the beam power was discarded to ensure clean cutting. Given that less than 2% of the total available power is used, a multi-beam approach can potentially increase processing speed even further.

5.2 Polymethyl Methacrylate (PMMA)

PMMA is a plastic which is widely used for a myriad of microfluidic applications (protein analysis, explosives screening, purine detection, etc. [80]). Various processing methods are used currently for the creation of microfluidic channels including thermal ablation. However, thermal ablation is limited to larger trench widths due to melt, a problem not encountered with high intensity nonlinear ablation.

Fig. 5.4 shows a square cut into PMMA with the high intensity laser. The square is cut with an x-axis galvo speed 537 mm/s, y-axis galvo speed 5.37 mm/s, 29 µJ pulse energy, 50 kHz repetition rate, and 20 passes on the slow axis giving a final depth of 300 µm. The edges of the square show no thermal damage and increasing the number of passes only contributed to a deeper feature. The depth of the feature was determined from OCT.

Sun et al. [32] demonstrates the creation of microfluidic channels using a CO$_2$ laser (thermal ablation) with a volumetric removal rate of 120 µm$^3$/µs. Here, the volumetric removal rate is estimated to be 217 µm$^3$/µs. It should be noted that the large surface area of this feature allows deeper cuts by translating the sample in the z-direction (i.e. moving the bottom of the feature back up to the beam waist) as it is now no longer limited by beam clipping, which is a problem for high aspect ratio features. The deeper cutting abilities was verified experimentally (not shown here). Thermal ablation also lead to highly tapered channels [32] while nonlinear ablation
Figure 5.4: Square feature cut in PMMA. The feature is created using galvo mirrors (see Fig. 3.1 b).

would have a more uniform bottom with less tapering along the edge.

Sun et al. also points out that thermal effects limited the channel sizes to be larger than 100 µm in width and is consistent with other groups who process PMMA with CO$_2$ lasers [81, 82]. Excimer lasers have been shown to overcome this limitation but require UV optics that are more difficult to integrate into machining platforms and/or ICI [83]. This is not a problem with high intensity nonlinear ablation. Fig. 5.5 shows trenches (channels) cut in PMMA with sizes suggested by collaborators from the Department of Chemistry at Queen’s University [84].

The debris seen along the edge is not thermal damage but recast from ablation. The thicker trench was cut with x- and y-axis galvo speeds of 0.48 mm/s and 537 mm/s, while the thinner trench used speeds of 537 mm/s and 0.24 mm/s for the x- and y-galvos respectively. Pulse energy was set to 25.5 µJ, with a repetition rate of 50 kHz. Both axes were moving at the same time resulting in 10 passes on the slow axis and 100 passes on the fast axis for each trench. This gave a final depth of
145 ± 5 µm as imaged through OCT, though an area where the two trenches overlapped is slightly deeper. This can be avoided with feedback and/or better motion control. Chen et al. describe various methods of fabrication for microfluidic channels, most of which take long periods of time [80]. Thermal laser ablation provides a faster alternative but is limited by melt effects. The work here demonstrates that nonlinear ablation provides a solution for fast fabrication of microfluidic channels which combines the versatility of a laser platform (as opposed to injection molding) with the ability to create small features due to its non-thermal nature.

5.3 Manual feedback with ICI

Especially with thermal ablation, laser micromachining can be a stochastic process leading to holes with varying depths for the same processing parameters. With an
CHAPTER 5. ABLATION OF OTHER MATERIALS AND FEEDBACK CONTROL

Figure 5.6: Side view of holes drilled in stainless steel a) without feedback and b) with feedback. A set number of pulses were used for a) while the number of pulses were varied for b). Note: b) is the same as Fig. 3.4.

in situ imaging technique, a closed feedback system can be applied and cut precision can be greatly improved over drilling with set parameters (i.e. same number of pulses for every hole).

The M-mode images shown in Chapter 4 demonstrate that the depth of the ablation front can be followed at rates of up to 300 kHz for the 805 nm imaging system. This leads to the possibility of ending machining once a desired depth is reached with up to 3 µs of temporal resolution. Fig. 5.6 shows earlier work using the 1320 nm
imaging system when manual feedback is applied in stainless steel. The control and feedback parameters are the number of machining pulses delivered and the final depth of the hole respectively, where the depth is measured with ICI.

Fig. 5.6 is a 3-D rendering acquired ex situ with the 1320 nm imaging system where the galvo mirrors in shown Fig. 3.1 b) allows the system to obtain tomographic profiles (see Section 3.2). The holes were drilled with the QCW laser at a repetition of 4 kHz and a pulse duration of 100 $\mu$s. Stainless steel has a short absorption depth for 1320 nm light ($\sim$28 nm, [85]) and thus the lowest depths seen in the figure represents the bottom of each hole. In Fig. 5.6 a), each hole was drilled with 250 pulses resulting in widely varied results and giving a mean depth of 320 $\mu$m with a standard deviation of 120 $\mu$m. This is purely due to stochastic variations (i.e. melt flow and re-solidification, recast) in the machining process. For Fig. 5.6 b), an initial 100 pulses were delivered and evaluated with in situ M-modes. Manual feedback was then applied by the author by varying the number of pulses to reach a target depth of 320 $\mu$m. This resulted in holes with a mean depth of 320 $\mu$m and a standard deviation of 10 $\mu$m giving an order of magnitude improvement in drilling accuracy. More recent work by the group has allowed the development of automatic feedback via LabVIEW and resulted in similar accuracy with much faster response times than manual feedback could provide [28].
Chapter 6

Conclusions and Future Work

With ICI, the work here has elucidated some of the underlying dynamics of ablation. The vast parameter space (pulse energy, pulse duration, repetition rate, etc.) open to laser micromachining setups lead to time consuming experimentation before optimization is found and ICI has helped to accelerate this process. Micrometre resolutions (transverse and axial) along with high imaging rates (>300 kHz for silicon linear arrays) allow for a better study of the ablation process. The versatility of the in situ technique and its optical nature makes it a natural counterpart to laser machining platforms and provides a mode for active feedback without the limitations of high cost.

Specifically, in studying the thermal ablation in silicon, ICI has allowed the characterization of melt relaxation times and the creation of a model that accurately predicts the ablation rate. The model allows ideal parameters to be narrowed down a priori without the need for (sometimes costly) experimentation and the ICI M-mode provides active feedback during ablation, allowing further improvements in precision. ICI and modeling has shown a material, silicon, exhibiting nonlinear thermal properties that can be exploited. By taking advantage of the rapidly rising absorption
depth with rising temperature, a cascaded absorption effect takes place and allows for ablation to occur at 1070 nm; an unusual wavelength to use due to silicon’s long absorption coefficient at room temperature. The model has shown that for thermal ablation, the important parameter for maximizing removal rate is not maximum instantaneous power or even average power, but pulse shape and duration. Changes in ablation rates were seen with varying pulse duration for constant energy, and pulse shaping showed the potential to aid or hinder material removal for a given duration and energy. Given traditional use of UV DPSS systems for silicon processing, this provides a more cost-efficient alternative in fibre laser technology and without the need of costly UV specific optics.

Where thermal ablation effects cannot be controlled, nonlinear ablation provides an alternative to applications that have traditionally shied away from laser micromachining. The cleaner ablation process allows not only the monitoring of the ablation front in some cases, but also the back interface of the material, allowing control of depth from that surface. This is important when through-holes are not desired and a priori information about the thickness of the sample cannot be obtained. When compared to the literature, nonlinear ablation provides faster processing times (two orders of magnitude in PZT for high frequency arrays) as well as the ability to create fine features (sub 100 µm wide trenches in PMMA, currently limited by thermal melt when processed using CO₂ lasers). Even though the full parameter space was not explored, the quality of the features shown demonstrate that a more detailed examination could set nonlinear ablation as the gold standard for fabrication.

Several avenues for future research arise from this work. For thermal ablation of silicon, the curious effect of a saturated ablation depth is not well understood. Possibilities include the high aspect ratio of the holes and beam clipping effects. Further investigation of this phenomenon will help to surpass this limit or utilize it to control
cutting. Also, while the modeling provides accurate results, a fitting parameter is undesirable and leads to ambiguity over the applicability of future predictions. Without moving to a 2-D model, an analytical solution should be found to determine $L_{\text{Radial}}$ more accurately.

ICI itself, while having demonstrated manual and automated feedback [27, 28], is still limited currently to tracking only the main ablation front. Further improvements could be made in tracking software to recognize sub-surface interfaces automatically. Processing time is also currently limited by complex calculations (i.e., linear interpolation, FFTs) to produce visual imaging data. By moving computations to dedicated hardware such as graphics processing units (GPUs), reaction times for feedback can be greatly improved. This is currently under investigation by the group.

The work shown here is only a glimpse of the potential of current and emerging laser manufacturing technology. This rapidly expanding field is only itself a small part of laser technology as a whole, which is rather curious for a ‘solution looking for a problem.’
Bibliography


