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The processing of SiC/SiC ceramic matrix composites using a pulsed Nd–YAG laser

Part I: *Optimisation of pulse parameters*

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The machining of a composite material comprising silicon carbide (SiC) fibres in a chemical vapour infiltrated SiC matrix has been investigated using a 400 W pulsed Nd–YAG laser. The principal aim of this work has been to determine, by comparison with the results obtained from other ceramic matrix composites optimum processing conditions for these materials with regard to both material removal rate and cut surface quality. Previous trials involving a borosilicate glass matrix composite and a magnesium–alumino–silicate glass–ceramic matrix composite, both incorporating the same SiC fibres, have highlighted the importance of the coupling of the matrix phase with the emitted laser radiation. The various phase's resistance to oxidative degradation at high temperatures is another influential factor. The material considered in this work has been shown to be particularly suitable in these respects, with the result that both cut rate and quality are significantly enhanced. Report is made of the effect of varying the laser pulse parameters such as pulse energy, duration and intensity and concentrates on the material removal rate. Part II of this work addresses the influence of *process* variables, such as choice and pressure of assist gas and the point of focus of the laser beam on the quality of the cut surface. © *1998 Chapman & Hall*

1. Introduction

The problems associated with the machining of ceramic matrix composite (CMC) materials have, in recent years become a focus of attention, necessarily preceding the widescale application of these materials. The currently established methods are limited largely to diamond grinding techniques, costly in both time and materials and often inducing damage that can significantly affect the in-service performance of the component. There is considerable potential for novel methods to revolutionize the processing of such materials, and a programme has been initiated to examine the various possibilities with an emphasis on laser methods.

The material studied is the third of a series of glass/ceramic matrix composites to be investigated as part of the overall programme. Previous reports [1–4] issued from this project have presented and discussed the results of trials performed on a borosilicate (Pyrex^{TM}) *glass* matrix composite (GMC) and a magnesium–alumino–silicate (MAS) *glass–ceramic* matrix composite (GCMC) respectively. Whilst both of these materials are reinforced with a SiC fibre identical to that used in the fabrication of the latest material, the major influences on the material removal rate and most notably the quality of processed surface is found to be the chemistry and the physical and optical properties of the matrix phase. The borosilicate matrix has been shown to be almost completely transparent to the 1.06 μm wavelength emission of a Nd–YAG laser and hence the machining of the GMC material is highly dependent upon the conduction of absorbed energy from the SiC fibres. The minor compositional changes in the system that produce the crystalline MAS matrix material also result in a phase that absorbs (to some extent, at least) the laser energy in its own right. This was reflected not only in the penetrating capability of the laser with the GCMC material, but also in the *quality* of the processed surface, primarily in terms of the reduction of redeposited, silicate glass dross.

The material introduced at this stage of the programme is different in many respects. Whilst still a composite structure, the matrix and reinforcing phases are essentially the same, i.e., silicon carbide, which has already been shown to absorb the laser wavelength more effectively than either of the previous matrix structures. The method of fabrication is completely different, employing chemical vapour deposition (CVD) and chemical vapour infiltration (CVI) techniques [5] instead of the lay-up and hot-pressing processes used to make the first two materials.

The pulsed Nd–YAG laser used for these trials permits a number of the pulse waveform parameters to be tuned, quite apart from the peripheral aspects that may be varied such as the type and pressure of assist gas that is employed. The previous work on the GMC material established a test programme that examined the influence of these parameters with a systematic approach. As with the MAS matrix material trials, and to further augment the findings of the total work to date, the trials on the CMC material have, wherever possible used the same methodology and parameter ranges. By careful comparison of the three materials that have been used as *specific* cases, trends may be identified that enable conclusions to be made about the processing of CMC systems in general. Once again, equal emphasis has been placed on optimizing both the material removal rate and the processed surface quality.

2. Material under examination

The tests have been performed on sheet specimens cut from a component form of the SiC/SiC material. The fibre used to produce this material is the proprietary Nicalon™ 201 grade SiC fibre, but instead of using continuous tows and filament winding them into unidirectional sheets, the fibre is supplied in a sheet of cross-weave mat. This is cut to size, and then a number of such lay-ups are cold pressed with an acrylic resin binder to produce the "green" form. Typically, twelve sheets of the cross-weave press to form a plaque approximately 3 mm thick. After the acrylic resin has cured (approximately 2 h) the solid form is put into a graphite mould. This is placed in a furnace at 500 °C to burn out the resin, after which the CVD operation is undertaken. Whilst confidentiality agreements restrict information on the exact details of this process or the resulting composite, a brief description of the general principles of its method of production are necessary as it differs significantly from the previous materials.

2.1. CVD and CVI manufacturing processes

Chemical vapour deposition (CVD) is used to deposit a layer of pure carbon on the surface of the fibres, which ultimately acts as an interfacial layer. This is a particularly critical step, as the interfacial layer determines many of the matrix-to-fibre bonding characteristics and therefore has a significant influence on the mechanical properties of the finished composite. This process is achieved by heating the fibre pre-form to approximately 1125 °C for 50–100 h in a low pressure methane atmosphere. The next stage is similar, but produces the matrix phase itself. This is termed chemical vapour infiltration or CVI, and is performed at approximately 1025 °C in a low pressure methyltrichloro silane atmosphere. This produces the SiC matrix, as well as HCl gas as a by-product. After the first infiltration run, the component is removed from the graphite mould, and further infiltration runs are performed to complete the production of the matrix phase. As a final process, the component form is encapsulated in a glass material. This is because the interfacial layer of carbon is prone to rapid oxidative degradation if exposed to air at high temperatures. A layer of glass becomes very soft well before temperatures rise to a critical level, and flows over any exposed fibres to seal the interfacial layer against such chemical attack.

The material produced by this method is relatively high in porosity, but precise in chemical composition. The use of similar phases for both the matrix and reinforcement has advantages in terms of relative thermal expansion coefficients and transmissivities, giving a composite with greater high-temperature capabilities [6] than either of the glass/ceramic matrix materials previously investigated.

3. Facilities and general laser configuration

The laser and control facilities used for these tests are identical to those used to process the previous materials to permit a valid comparison of the results to be made. All of the trial sets have been performed with the Lumonics JK701 Nd–YAG laser. It has been used in the (low divergence) LD2 resonator arrangement, the major performance characteristics of which are listed in Table I. This was chosen in preference to the LD1 resonator which operates at a lower average power, and the welding resonator, which whilst permitting greater variation of average power, does so at the expense of beam divergence. This would limit the output beam's intensity, which is undesirable when attempting to investigate maximum rates of material removal. As one of the main considerations of the test methodology has been to ensure that all sets of test data bear comparison with regard to as many parameters as possible, the LD2 resonator was selected as providing the greatest flexibility.

An 80 mm focal length cemented achromat was used for the final stage lens. The JK701 laser has an internal power meter, but this takes its measurement from a point just after the resonator and before the final optic train, including the beam expansion telescope (BET) and the final lens. In addition, at extremes of adjustment it is possible to produce "clipping" of the beam by the nozzle assembly, so an external power meter – a water-cooled Coherent "Fieldmaster" was used to verify the internal meter's reading and evaluate the power losses occurring in the final optical stages.

A custom-built nozzle assembly has been manufactured that facilitates nozzle alignment, allows rapid access to replace marked or damaged cover-glasses as well as permitting full adjustment of nozzle stand-off distance independently of focus position for trials involving changing the focal-point-to-surface distance, assist gas variations etc. This has made adjustment of the laser set-up an easier task than would be required if the equipment were used for a standard industrial application.

Positioning of the workpiece was achieved by the use of Unidex 400 CNC controller connected to a 3-axis workstation. This fully-programmable facility

TABLE I Performance of the laser in the LD2 configuration

Parameter	Range (LD2 resonator)
O/P wavelength	1060 nm
O/P energy (approx.)	0.5–15 J variable
O/P power (approx.)	tuned cavity 230 W nom.
Repetition rate	17–200 Hz variable
Pulse duration	0.5–5 ms variable

allows $600 \times 600 \times 300$ mm of accurate (± 0.2 mm over range) and repeatable travel, facilitating the duplicating of test geometries on different specimens.

Oxygen, nitrogen, argon and air were investigated as choices of assist gas. These were manifolded to allow a maximum supply pressure of approximately 8×10^3 Pars, measured from a point close to the nozzle orifice.

4. Test methodology

The first series of tests investigated the effect upon the material removal efficiency of changing the laser pulse duration (whilst holding the pulse energy constant), changing the laser pulse energy (whilst holding the pulse duration constant) and changing the beam intensity, at pre-determined constant levels of pulse energy *and* duration (and hence at constant peak powers).

It should be remembered that within the pulse energy and duration trials, the average power and peak power of the laser pulses are *not* held constant; they change in accordance with the varied parameter whether that is pulse energy or pulse duration, according to the relations;

$$\text{Average Power (W)} = \text{Pulse Energy (J)} \times \text{Repetition Rate (Hz)} \quad (1)$$

$$\text{Peak Power (kW)} = \text{Pulse Energy (J)} / \text{Pulse Width (ms)} \quad (2)$$

To isolate the effect of the tuned condition being achieved by varying the pulse repetition rate, the majority of these tests were performed in the single-shot mode. These produced blind holes which could be simply sectioned for analysis. To assess the volume of material removed in each case, it was noted that the geometry of these holes was quite consistent. It was determined therefore that the most reliable and simple measurement to take was the *depth* of the blind hole, and consider this proportional to the hole volume. With previous tests having highlighted the inconsistency of the lasing process with this type of material, six repeated tests were performed for each parameter condition to give an indication of the degree of scatter of results for nominally identical lasing conditions.

5. Laser processing results

5.1. Pulse duration variation

Six individual, single-pulse, blind holes were drilled for each of a range of pulse durations whilst maintaining a constant pulse energy. This series of trials were then repeated for two other levels of pulse energy. The pulse energy levels were 3.15, 7.25 and 14.5 J. The holes were then sectioned for analysis in the scanning electron microscope.

Fig. 1 is a graphical representation of the results, with the variation in pulse duration plotted against the depth of hole produced. Over the whole data set there is a general increase in the penetration of the plaque as the pulse duration is lengthened, although

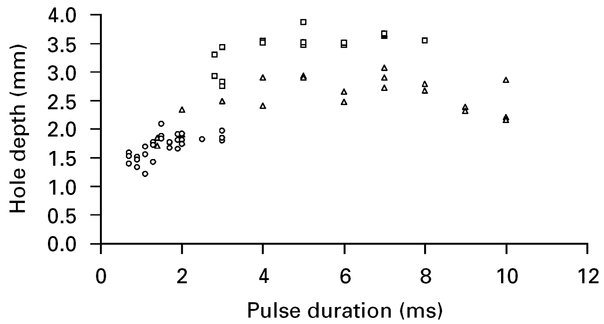

Figure 1 Effect of pulse duration at constant pulse energies of (□) 14.5 J, (△) 7.2 J and (○) 3.15 J.

the widest data set, that for the pulse energy of 7.2 J shows evidence of reaching a penetration limit. There is a significant improvement in material removal efficiency as the pulse energy is increased (compare the points as plotted at 3.00 ms, where the three data sets overlap). As the peak power of the incident laser pulse is given by Equation 2 above, this suggests that of the two parameters, the pulse energy component of the peak power is the stronger influence on material removal. This is in agreement with the conclusions of trials performed on the GMC and GCMC materials.

Direct comparison with the results from the processing of the previous materials (shown schematically in Fig. 2) illustrates a marked increase in the penetrative capabilities of the laser with the SiC/SiC composite. This ranges from approximately 50% at the lower pulse energies, to over 75% at 14.5 J. There is, however a greater degree of scatter in the results, particularly when compared to those for the MAS matrix material. This is probably a result of the greater degree of random porosity exhibited by the SiC/SiC composite.

5.2. Pulse energy variation

The pulse energy investigation was conducted in a similar manner, but with the pulse energy varied between its tuned limits at each of three pulse width values, 0.6, 1.5 and 5.0 ms. The resulting depth-of-hole measurements are plotted in Fig. 3.

The overall trend of Fig. 3 is that the penetration of the single laser shots increase, both within the data sets of increasing pulse energies and with the incremental increases in pulse duration. There is once again evidence that an upper limit is reached at the highest peak powers, although this may reflect more upon the ability of the assist gas to "flush" the vapourised material than the power of the laser itself at this depth.

Comparison with the corresponding results from the previous trials on the GMC and GCMC materials (shown schematically in Fig. 4) show that there is a very great increase in the material removal efficiency, especially at the higher peak powers where an increase of over 100% is demonstrated. The scatter of the data points is generally low for this series of tests, with the possible exception of the trials performed with a 1.5 ms pulse duration, but all data points lie

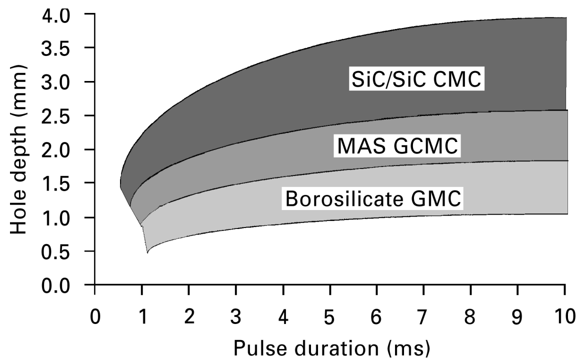

Figure 2 A comparison of the effect of the pulse duration at constant pulse energies for the three types of materials investigated.

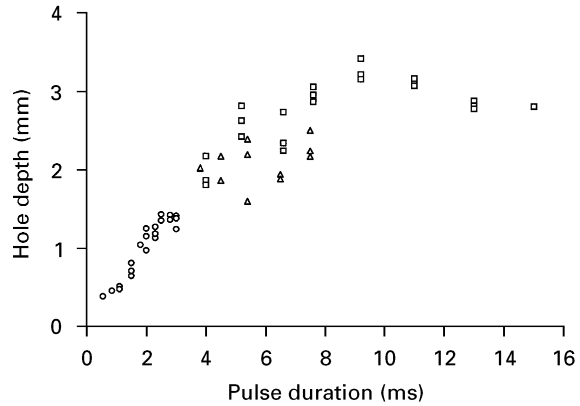

Figure 3 Effect of pulse energy, at constant pulse durations of (□) 5 ms, (△) 1.5 ms and (○) 0.6 ms.

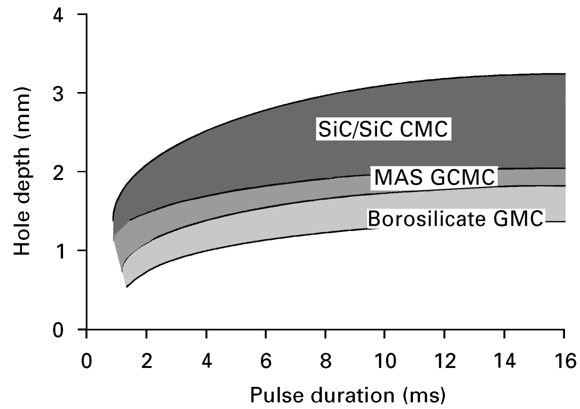

Figure 4 A comparison of the effects of the pulse energy at constant pulse durations for the investigated materials.

within a trend that clearly shows an improvement in material removal in comparison to the previous composites studied.

5.3. Peak power variation

To aid a full comparison with the previous trials, the results of the pulse parameter tests are plotted as penetration versus peak power, Figs 5 and 6 for the constant pulse energy and constant pulse duration respectively.

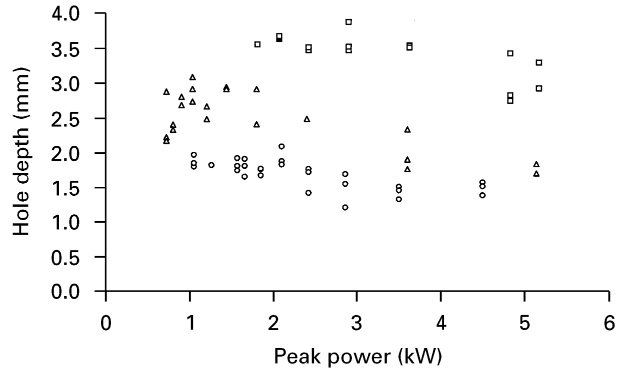

Figure 5 Peak power versus hole depth for varied pulse durations. Key: (□) penetration at 14.5 J, (△) penetration at 7.2 J and (○) penetration at 3.15 J.

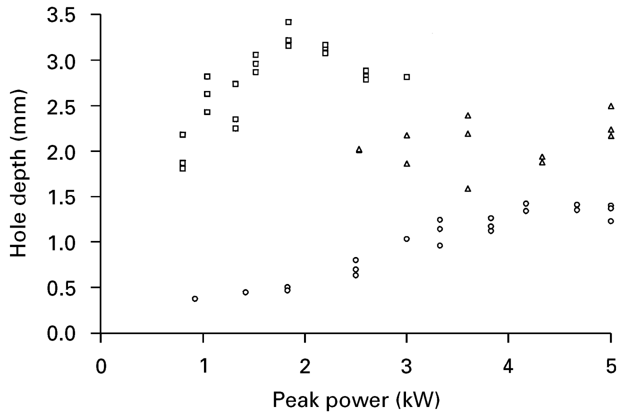

Figure 6 Peak power versus hole depth for varied pulse energies. Key: (□) penetration at 5 ms, (△) penetration at 1.5 ms and (○) penetration at 0.6 ms.

The results as depicted in Figs 5 and 6 are in good agreement with the previous tests and contribute more evidence to support the conclusions made in those instances; that the penetration is increased by raising the pulse energy but increasing the pulse duration other than from the lowest levels has little effect. Although the greater scatter in the current sets of data would make it difficult to justify these findings on their own, the trends are similar in each case. Fig. 5, the results for three levels of constant pulse energy, shows three quite distinct strata of data, each with very little if any increase in penetration with increasing peak power. The major contribution to the increasing level of material removal would seem to be confirmed as the pulse energy. There is a 50–100% increase in the extent of penetration when compared with the MAS matrix material trials. The data represented in Fig. 6 once again shows distinct strata in the plotted points (although in this case the scatter of points is more severe, especially for the higher pulse durations) but in each case, there *is* evidence of an increase in the depth of hole as the peak power is raised. As the pulse energy is the parameter that is *causing* the increase in peak power when the duration is held constant, this must indicate a greater dependency on energy than duration. This is in complete agreement with the GMC and GCMC trials, despite the differences in the materials under test [1, 3].

5.4. Pulse intensity variation

As far as is permitted by the tuned operation of the laser, corresponding values for the laser parameters were used for the investigation of the affect of changing the beam intensity. By using the beam expansion telescope (BET) to make this adjustment, these results may be directly compared with those of the previous tests. The results are presented in Fig. 7.

As with the results obtained for the plots of the pulse parameters versus peak power, it would be difficult to draw any convincing conclusions based solely on the data obtained from the intensity trials on the SiC/SiC material. This is primarily because of the scatter in the results making a clear trend in the data unclear. However, the results as shown in Fig. 7 do bear comparison with the equivalent data from the two previous material's trials [1, 3]. In those tests, an inflexion in the trend was noted, giving a maxima in the material removal efficiency at approximately 1.7×, and a minima at approximately 2.2×. With these results in mind, there *is* some confirmation in the latest set of data; certainly the penetration is uniformly greater at 1.5–1.7× than at 2.3×.

6. Scanning electron microscopy examination and discussion of results

6.1. Pulse width/energy, peak power variation tests

The blind holes produced by each of the series of tests investigating the effects of varying the pulse duration, energy and intensity were examined to determine any difference in the resulting hole geometry or extent of material damage.

The first point worthy of note is a general difference in the shape of the drilled holes. In the case of the GMC material, the hole geometry depended upon the extent of penetration. Holes less than 500 μm deep were convergently tapered, but subsequent penetration resulted in a parallel bore; "established" holes are invariably pencil-sectioned. A similar result was found with the MAS matrix GCMC, but in addition it was noted that the bottom of the blind holes were generally more pointed than their pyrex-matrix counterparts. This situation would seem to be taken to the extreme in the case of the SiC/SiC trials, as the holes maintain a convergent taper to the limit of penetration, even in deep-drilling (up to 4 mm deep). Fig. 8 shows such a hole.

It has been stated that the borosilicate glass matrix is almost completely transmissive of the 1.06 μm wavelength of the Nd–YAG laser, and that matrix vaporization is therefore almost entirely due to conduction of heat from the more absorptive SiC fibres. The MAS matrix is less transparent to the laser, resulting in a greater material removal efficiency of the composite as a whole and differences in the geometry of holes and kerfs. The more pointed shape of the blind holes, initially at the ends of the MAS trials but in the entire hole in the SiC/SiC examples may be a result of primary removal of the matrix, rather than secondary, i.e., as a result of heat conduction from the fibres. In this case the vaporization front could possibly assume the shape of the energy distribution across the diameter of the laser beam (which is ideally a Gaussian profile), rather than a more spherical, conduction-induced shape. Internal reflections of the incident laser beam and the various phases reaction to these may also account for the differing hole geometries.

There is a great deal less evidence of redeposited material than was found with either the GMC or GCMC materials, either on the surface around the entry hole or on the bore of the hole itself [1, 3]. Fig. 9 (a–c) shows how clearly the processed fibres are exposed in the almost complete absence any deposited material as found in the previous material's trials. There is also very little evidence of the "countersink" feature around the entrance hole that was found with the previous composites, where a region showed depletion of the matrix and debonded fibres to a depth of about 200 μm and diameter up to 900 μm [1].

6.2. Pulse intensity variation tests

The maxima/minima feature in the penetration exhibited by the various trials and corroborated by Fig. 5 is of interest on two counts. Firstly in terms of the implications; the fact that a maxima (which relates to a greater material removal efficiency) appears at a setting of approximately 1.7× beam magnification, rather than at the highest beam intensity as might be expected, suggests an experimentally derived optimum

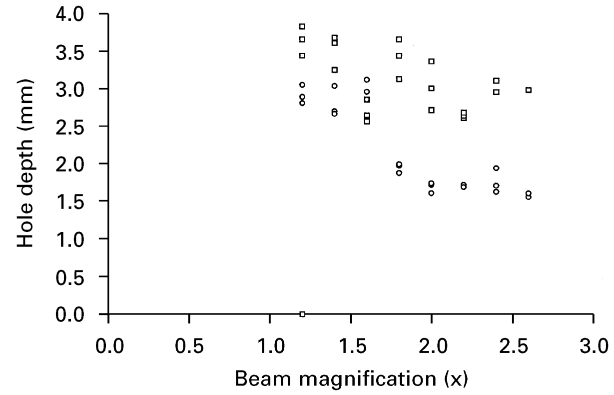

Figure 7 Effect of pulse intensity. Key: (□) 14.5 J, 5 ms and (○) 3.15 J, 0.9 ms.

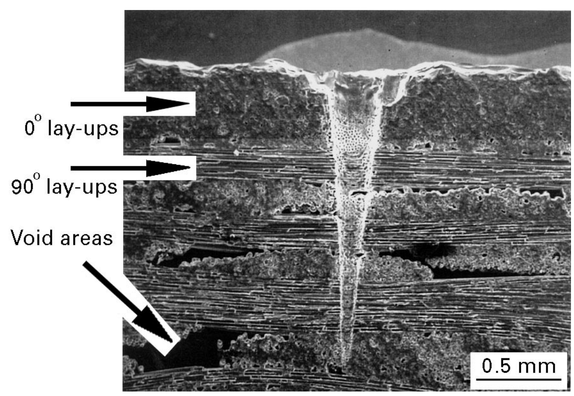

Figure 8 Constant-taper, deep-drilled hole.

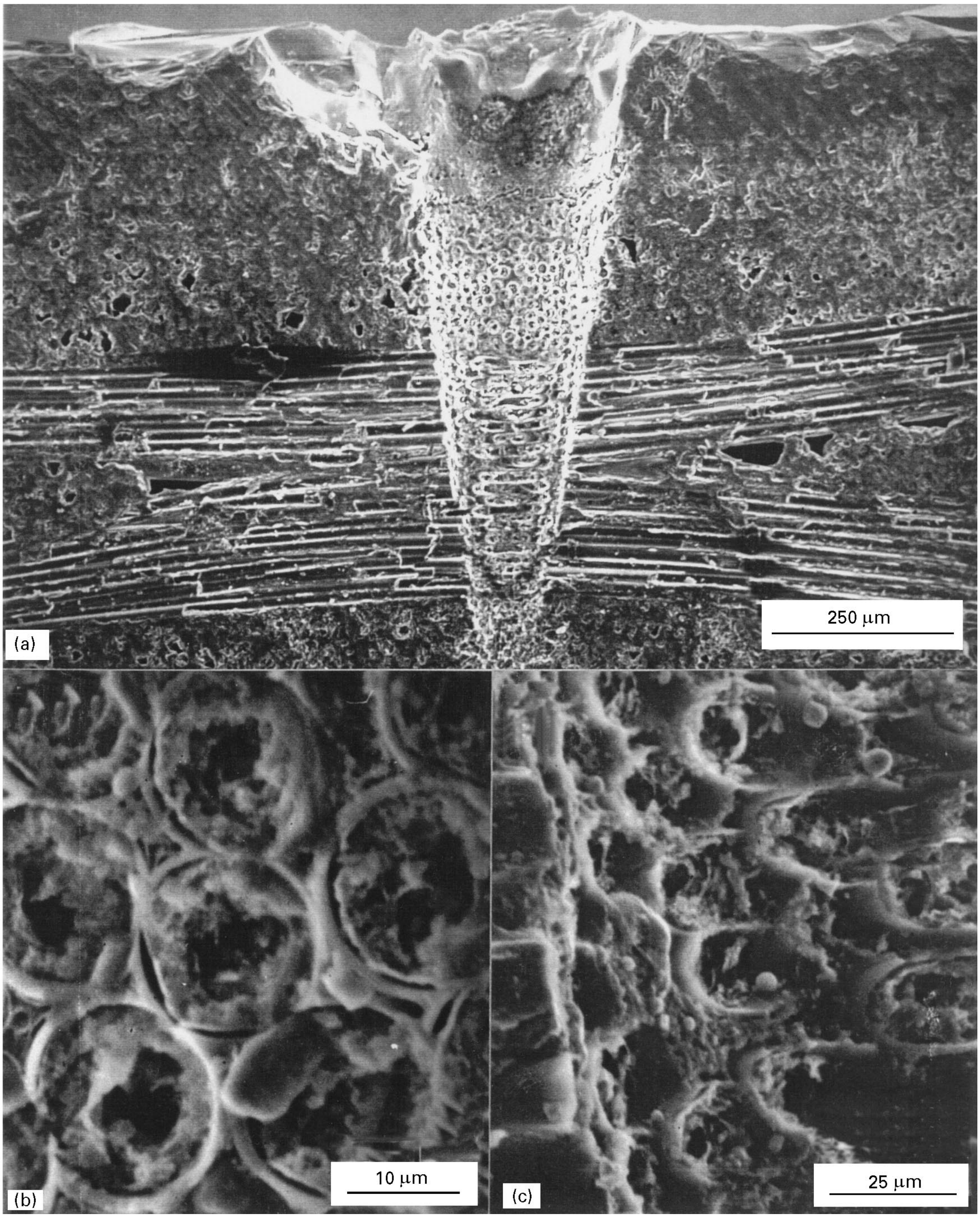

Figure 9 (a) showing general geometry of hole: note the lack of redeposited material in the bore of the hole and the cleanliness of the entrance hole. (b) Through-ply, and (c) cross-ply fibres exposed by the processed hole. Note the evidence of the pure carbon interfacial layer around each fibre.

setting for this parameter. Secondly, the difficulty in explaining satisfactorily why the trials show both a maxima and a minima at all, and at approximately the same values irrespective of pulse energy settings. Similar effects have been noted when processing other materials, notably metallics [7] but a definitive justification is elusive. Matsunawa *et al.* [7] have considered whether it is a result of beam/material interaction; for instance, the molten material particles varying in size, and therefore absorbing the incident beam to differing degrees. This must remain an explanation of some conjecture, especially as it has been determined that the majority of material is removed in the vapour phase; particulate theories in these circumstances must be suspect. It has already been speculated that this is a characteristic of the laser optic system (as it seems to be quite independent of the material being processed), and these results would, if anything, support this conclusion.

7. Summary

This study investigates the YAG laser machining trials of a SiC fibre reinforced, SiC (CVD/CVI deposited) matrix composite. The test conditions and parameters have, whenever practical, copied those performed on two glass/glass–ceramic matrix, SiC fibre composites prior to this work, to aid comparison between the results.

The work has again tried to address both the question of optimizing the material removal rate with the available equipment and the factors that influence the quality of the processed surface. The latter forms the subject of the second part of this work.

In common with the conclusions of the work on the Pyrex and MAS matrix composites, the pulse parameter trials indicate that there is an increase in the material removal efficiency associated with increasing the incident peak power, and of the two parameters that influence the peak power level, it is the pulse energy rather than the pulse duration which makes the major contribution to this result. Of the two previous materials, the MAS matrix produced higher levels of material removal for any given set of laser pulse parameters, a feature that was attributed to the fact that the pyrex matrix is almost completely transparent to the Nd–YAG laser's emission wavelength, whereas the MAS matrix is able to couple with the laser energy to some extent. The implication in this is that to be able to process the pyrex composite *at all*, the matrix must be removed by heat transmitted from the SiC fibre phase. It is not surprising then that a composite in which both the matrix and the fibre are SiC is able to absorb the laser's energy more effectively, resulting in significantly higher material removal rates for any given set of pulse parameters.

The drilled holes in the SiC/SiC material exhibit a much cleaner, more "precise" processed surface than had been seen in the previous work, the trials at this stage all using nitrogen as the assist gas. In the second part of this study, the influence of varying both the type and pressure of the assist gas is reported.

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