

## Chapter 11: Discussion

### 11.1 Interpretation of results

#### 11.1.1 Green processing

Although other methods of green processing, i.e. dry pressing, slip casting and centrifugal casting, were not used in this work, literature shows that filter casting is the preferred green processing route for the processing of nanocomposites (at least in a laboratory environment). Dry pressing generally gives compacts of poorer quality than 'wet' processing routes, while slip casting and centrifugal casting is impractical compared to filter casting.

Some of the practical problems with filter casting presented in literature may not have been noticed in this work. These are recovery strain (samples were too small to observe any recovery) and separation of particle sizes (powders used had fairly narrow particle size distributions). Separation of the suspensions components (diamond and alumina) during filtration would have been discovered if it did occur, but were not observed as long as fresh (not older than c. 6 h) suspensions were used.

In the mercury porosimetry run (section 7.1, page 52) the greatest frequency of pores occurred at pore sizes of c. 50nm, which is of the same order of size as the particle. This is to be expected of fairly monosized particles, since the size of voids between particles would be of the same order as the size of the particles. No peaks occurred at larger pore sizes, indicating that no large cracks were present.

#### 11.1.2 Firing

The sintering results of Yeh and Sacks (100 % relative density at 1 150 °C in air with AKP-50, section 2.2, page 19) could not be duplicated. With similar conditions 99 % relative density could only be obtained at 1400 °C (fig. 7-2, page 53). The addition of titania improved the situation, with 99 % relative density occurring at 1300 °C.

It is difficult to compare HIPping results of this work with HIPping results from literature (page 26), as the starting conditions (green compact preparation history and especially pre-HIP heat

treatment history) of compacts are not comparable. Nonetheless HIPping conditions used in this work (section 6.4.3, page 51) does not seem to be too excessive compared to those from literature (page 26).

### 11.1.3 Graphitisation

Although it is impossible to obtain reliable quantitative results from Raman spectroscopy without extensive research into the specific case, there should at least be some relationship between peak intensities and the degree of graphitisation. For instance, smaller intensities at the diamond wavenumber and higher intensities at the graphite and amorphous wavenumbers might correspond to a higher degree of graphitisation. Features on the spectra obtained (fig. 7-3, page 55) and the spectra of Knight and White (1989) (fig. 4-6 to 4-9, page 34) are compared in table 11-1.

The 'bulge' to the right of the diamond peak (indicated in fig. 7-3) might be either attributable to the formation of amorphous carbon (typified by a weak peak at c. 1350, compare fig. 4-8) or due to a possible shift on the diamond peak in the presence of alumina (compare fig. 4-9). Since the 'bulge' is more prominent with the samples HIPped at 1 250 °C, where one might expect less graphitisation and less diamond to amorphous carbon transition, it could rather be indicative of the closeness of contact between diamond and alumina (similar to that occurring with vapour deposited diamond on alumina) in these samples, than the presence of amorphous carbon (unless amorphous carbon also forms during vapour deposition of diamond on alumina, although this possibility was not mentioned by Knight and White).

The slight 'bump' occurring with all samples at a wavenumber of 1580 is very likely due to formation of graphite (which has a peak at 1580, compare fig. 4-7).

There is evidence for graphitisation in all samples, but it is difficult to rank samples according to degree of graphitisation. For instance, on the evidence of the strength of the diamond peaks, it would seem that 15 $\diamond$ - $\alpha$ -CP-H1400 (i.e. the sample with the hottest HIPping) experienced the most graphitisation but it also has the one of smallest graphite 'bumps'.

**Table 11-1: Comparison of Raman spectra.**

		Feature →	Diamond peak	'Bulge' on the right of the diamond peak	Graphite 'bump'
		Wavenumber →	1 332	1 332-1 380	c. 1 580
		Mode of comparison →	Intensity	Ranking	Intensity
HIPping temperature (°C)	Preparation route				
1 400	CP		4 000	4th	310
1 300	pH $\alpha\phi$		11 000	3rd	310
1 250	pH53		7 000	2nd	1 100
1 250	HP		16 000	1st	600

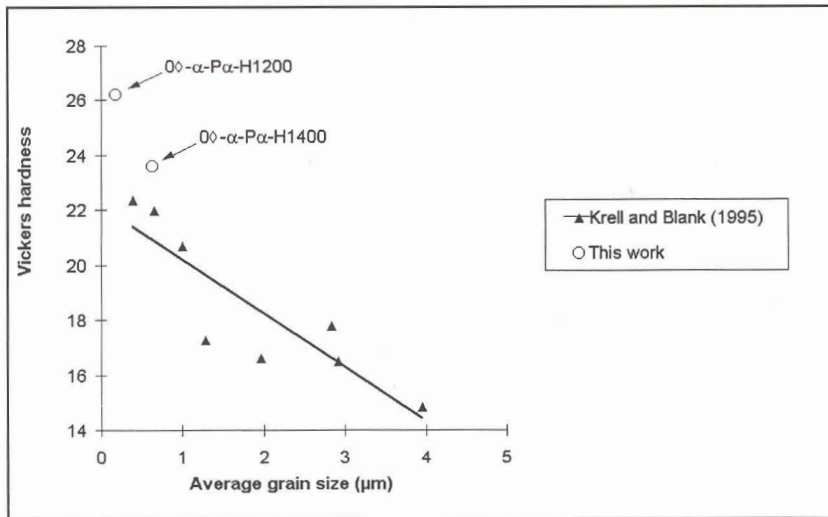
#### 11.1.4 Fired microstructure

The general quality of compacts are good, as far can be seen from fracture surfaces. Alumina grains are small and size distributions are narrow. Diamond particles are well dispersed throughout the examined samples, with the exception of the grouping seen in 15 $\phi$ - $\alpha$ -pH $\alpha\phi$ -H1300 (fig. 8-10, page 62), which is almost certainly due to diamond agglomeration carried over from green compact formation. Fig. 8-23 and 8-24 (page 66) shows interfaces between diamond and alumina that are characteristic of all samples: The diamond particles are seen to fit closely into the alumina matrix. No obvious graphite could be distinguished on diamond surfaces, even though it must have been present as shown by the Raman spectroscopy.

As can be expected, grain size increases with HIPping temperature (fig. 8-24, page 67). The presence or absence of diamond would not seem to have an effect on grain size, as the alumina grain size in non-diamond containing samples followed the same trend as the diamond containing samples (fig. 8-24). Not enough data are available to draw conclusions as to the effect of the green processing route.

#### 11.1.5 Hardness and toughness

In non-diamond containing samples, hardness was slightly above the relationship with grain size given by Krell (1995) (fig. 5-6) as shown below in fig. 11-1.



**Fig. 11-1:** Hardness (at 10 kg f) of alumina of this work compared with Krell and Blank's (1995) values.

Whereas in the work of Noma and Sawaoka (1984 and 1985) hardness decreased with the incorporation of diamond (fig. 5-11, page 46), in this work there was no significant change with the incorporation of diamond. Hardness varied between 22 and 27 GPa (table 9-1, page 71) regardless of diamond content. This is an improvement on the values reported by Noma and Sawaoka (20 GPa and less, fig. 5-11). Hardness of samples fabricated in this work compares favourably with that measured for LX11 (26 GPa). As hardness did not differ much between samples,  $K_C^{3/4}H_V^{1/2}$  is in effect an indicator of toughness in this work.

The measured hardness values of this work are probable relatively accurate (subject to the error discussed in section 9.1 on page 68) as are the toughness values of the non-diamond containing samples. However, it is likely that the toughness of diamond containing samples were overestimated. Diamond containing samples could not be polished to the same surface finish as the non-diamond containing samples (compare fig. 9-6, page 74 with fig. 9-8, page 75) with the result that the measured cracks lengths could have been shorter than actual crack lengths. (Note again, however, that micrographs like those of fig. 9-3 to 9-8 were not used for crack length measurement, measurements were made on the PC screen.) Although the toughness of diamond containing samples would appear to be higher than those of the non-diamond containing samples, with toughness of non-diamond containing varying between 1.5 and 2.1  $\text{MPa}\cdot\text{m}^{3/2}$  and diamond containing samples varying between 2.7 and 4.2  $\text{MPa}\cdot\text{m}^{3/2}$  (table 9-1), this apparent toughening effect of diamond should therefore be interpreted with caution. At any rate, toughness obtained in this work and toughness obtained by Noma and Sawaoka (1985) cannot be compared directly as the toughness of the respective unreinforced matrices differ (probably

mainly due to the difference in matrix grain size in this work being  $0.6 \mu\text{m}$  and less while that of Noma and Sawaoka is in the order of  $1.5 \mu\text{m}$ ): In this work toughness obtained with unreinforced samples varied between  $1.5$  and  $2.1 \text{ MPa}\cdot\text{m}^{1/2}$  while that of Noma and Sawaoka is  $4.5 \text{ MPa}\cdot\text{m}^{1/2}$ . However, the maximum improvement in toughness obtained by Noma and Sawaoka is  $57 \%$  (see fig. 5-12, page 45) while, in this work,  $15\phi\text{-}\alpha\text{-CP-H1400}$  has double the toughness of  $0\phi\text{-}\alpha\text{-P}\alpha\text{-H1400}$  (table 9.1). As  $15\phi\text{-}\alpha\text{-CP-H1400}$  was certainly not graphitised as much as the samples in Noma and Sawaoka's work, this puts the validity of graphitisation as the mechanism for the toughening effect by Noma and Sawaoka in some doubt, although, again differences in alumina matrices may invalidate such comparisons.

There are no trends that can reasonably deduced between HIPping temperature or grain size and  $K_C^{3/4}H_V^{1/2}$ , or as can be seen below in fig. 11-2 and 11-3.

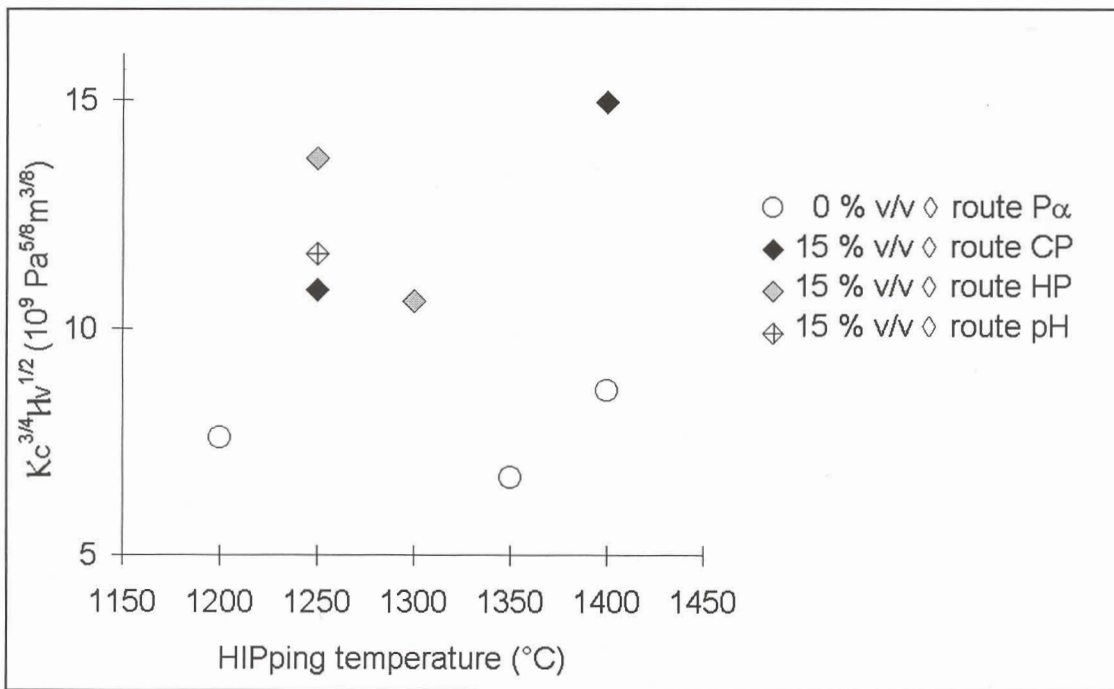


Fig. 11-2: The abrasion resistance indicator,  $K_C^{3/4}H_V^{1/2}$ , as a function of HIPping temperature.



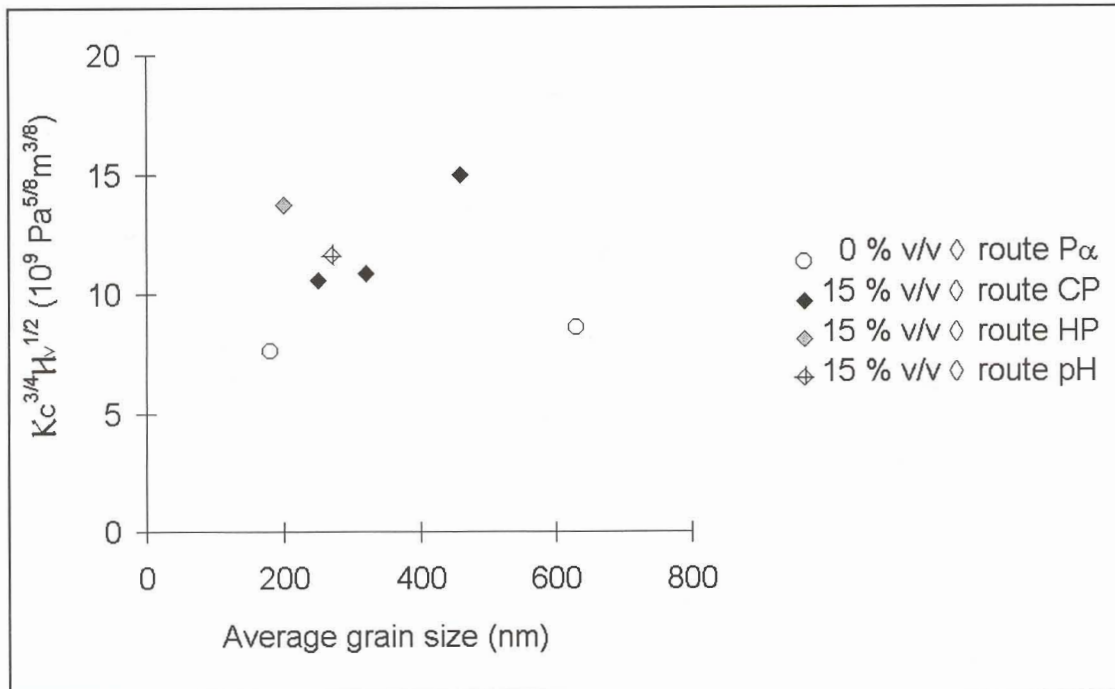


Fig. 11-3: The abrasion resistance indicator,  $K_c^{3/4}H_v^{1/2}$ , as a function grain size (as defined in section 8.1.3).

### 11.1.6 Wear

As the 'conclusive' characteristic, the wear performance can depend on many processing parameters or intermediate properties (see fig 1-2, page 3).

No simple relationships exist between HIPping temperature, grain size or green preparation and wear performance; data are randomly distributed for these relationships, which could indicate that experimental conditions during manufacturing of samples were not controlled tightly enough.

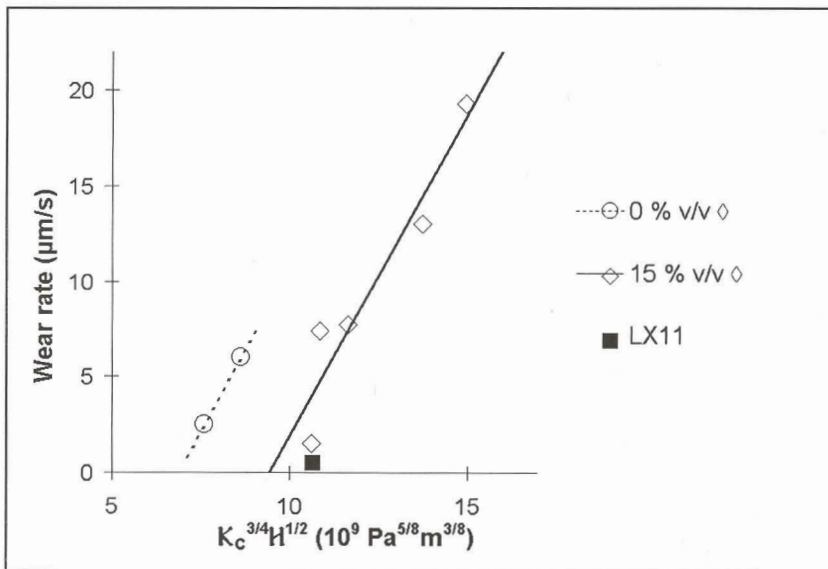
Sample 15 $\diamond$ - $\alpha$ -pH-H1300 showed more grouping of diamond particles (fig. 8-10, page 62) than other samples but exhibited the lowest wear rate. This behaviour is not what should be expected, but its low wear need not be connected to its lack of diamond dispersal. On the other hand, no other explanation can be given for this behaviour.

Even more peculiar is the relationship between  $K_c^{3/4}H_v^{1/2}$  and wear rate. Whereas  $K_c^{3/4}H_v^{1/2}$  is supposed to indicate resistance to abrasive wear (section 5-5, page 40), the wear rate actually increases with increasing  $K_c^{3/4}H_v^{1/2}$ , as can be seen below in fig. 11-4. Stated differently, this means that the tougher and the harder samples are, the faster they wear.

Some possible explanations for this are the following:

- As  $K_C^{3/4} H_V^{1/2}$  indicates abrasive wear resistance, this might mean that the dominating wear mode is not abrasive, which in itself would be surprising.
- Either the measurement of wear rate or toughness (as already pointed out hardness does vary much between samples and  $K_C^{3/4} H_V^{1/2}$  is in effect only an indicator of variation of toughness in this work) is suspect.
- Some other property of the wear testing system has a larger influence on the wear rate than toughness.

An additional result from fig. 10-7 is that, if the limited data for unreinforced alumina are extrapolated, it would seem that diamond reinforced alumina would have some advantage above unreinforced alumina of similar  $K_C^{3/4} H_V^{1/2}$ . However, this could be due to the overestimation of the toughness of diamond containing samples already discussed.



**Fig. 11-4:** The wear rate compared to the abrasion resistance indicator,  $K_C^{3/4} H_V^{1/2}$ .

## 11.2 Conclusion

It would not seem from this research that alumina-diamond would be a feasible material for low wear applications.

This is especially clear from wear tests, in which LX11 outperformed diamond-alumina. Of course, the wear testing method was very aggressive compared to other methods, but it is still unlikely that diamond-alumina would compare better under other wear testing conditions. Even if diamond-alumina can be optimised to compare with something like LX11, or in the unlikely case of improving on it, it is dubious if production would be economical. A cutting tip like LX11 currently sells commercially at a price in the order of R50. It is doubtful if diamond-alumina tips could be produced at such prices, especially with the complicated 'wet' processing involved. Of course, due the difficulty of preparing samples, most test work was done on a relatively few samples. Even if more exhaustive research is done, though it is still unlikely that the overall evidence would change.

However, the emphasis in experimental work in this thesis has been on efforts to limit graphitisation. Graphitisation was perceived as a barrier to adhesion between diamond and alumina. In contradiction with the approach in this work, Noma and Sawaoka (1984 and 1985) actually deliberately graphitised the diamond particles in their composites and claimed this improved toughness. If one would still want to pursue the possibility of diamond-alumina this effect should be looked into.

Furthermore, if only the lower (and conservative) asymptotic mixing law (equal wear mode) of Axén and Jacobson (1994) (fig. 5-4, page 39) is considered, it would seem that the true advantage of composites only comes at much higher concentrations of the harder phase than the 15 % v/v used here in this work. This leaves the possibility of higher diamond concentration leading to better properties than those experienced in this work with its low diamond concentration. One might even imagine alumina merely having a role of only cementing diamond particles together.