Chapter 8: Fired microstructure

8.1 Introduction

8.1.1 Lack of micrographs of polished and etched samples

Ideally, work of this nature should also include images of polished and etched surfaces, which should give a representative image of grain size. Unfortunately, this was not possible. Table 8-1 gives the steps used in surface preparation for each sample that was used with some success on both diamond containing and non-diamond containing samples. Fig. 8-1 and fig. 8-2 show the finishes obtainable with this procedure. Thermal etching would have damaged the diamond particles and chemical etching did not achieve the desired effects: Chemical etching for 20 minutes with 40 % w/w hydrofluoric acid as recommended by Petzow (1976) produced the selective etching seen in fig 8-3. For these reasons only fracture surfaces are presented here.

***Table 8-1**: Surface preparation steps.*

Time (minutes)	Particle type	Particle size	Surface	Surface speed (cm/s)
~ 1	SiC	180 grit	Sanding paper	< c. 120
~ 1	SiC	220 grit	Sanding paper	< c. 120
~ 1	SiC	320 grit	Sanding paper	< c. 120
~ 1	SiC	400 grit	Sanding paper	< c. 120
~ 1	SiC	600 grit	Sanding paper	< c. 120
~ 1	SiC	800 grit	Sanding paper	< c. 120
5	Diamond	9 μm	Polishing cloth	< c. 105
5	Diamond	3 μm	Polishing cloth	< c. 105
5	Diamond	1 μm	Polishing cloth	< c. 105

8.1.2 Identification of diamond

Diamond particles can be distinguished as micron-sized particles in the micrographs, but they are also identified in black on the maps attached to micrographs showing diamond containing samples.

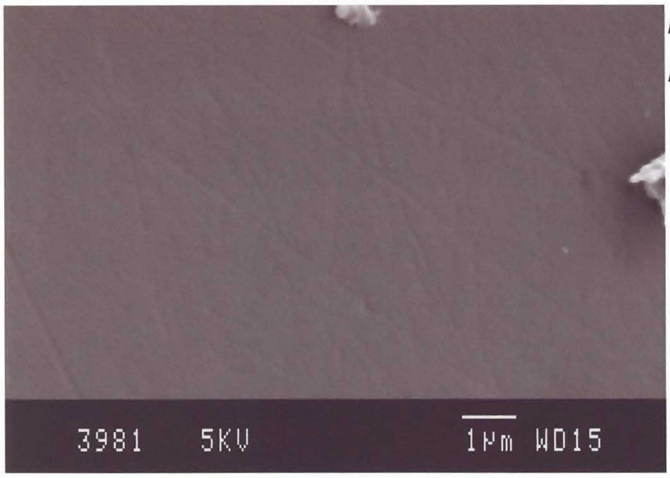


***Fig. 8-1**: A non-diamond containing sample (0φ-α-Pα-H1400) after polishing and before any etching.*

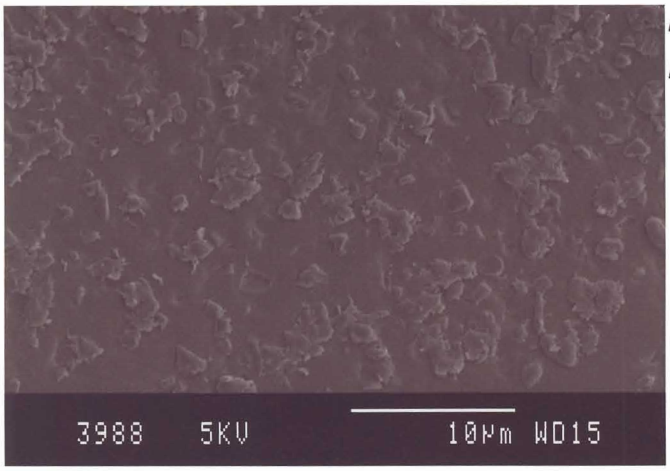


***Fig. 8-2**: A diamond containing sample (15φ-α-CP-H1400) after polishing and before any etching.*

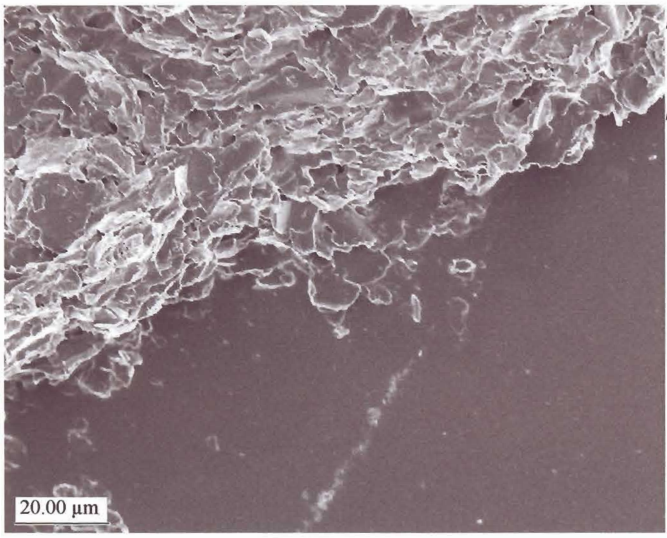


***Fig. 8-3**: The same as in fig. 8-1 after chemical etching. The etchant attacked the area in the upper half of the micrograph while area in the lower half is unaffected.*

8.1.3 Grain size estimation

By counting the number of times a line across a micrograph of a fracture surface intersects grains a crude estimation of grain size can be made. An average grain size $\bar{d}_{grain}$ would then be given by

$$\bar{d}_{grain} = \frac{[\text{Length of line}]}{[\text{Number of grains}]} \qquad (8\text{-}1)$$

As an example, consider fig. 8-4. The line spans 4.0 µm and crosses the 20 grains indicated. The average grain size for this sample is therefore 200 nm. (Ideally estimation should have been performed with properly polished and etched surfaces as, for example, per ASTM E 112-82. This was not possible for reasons already explained in paragraph 8.1.1.)

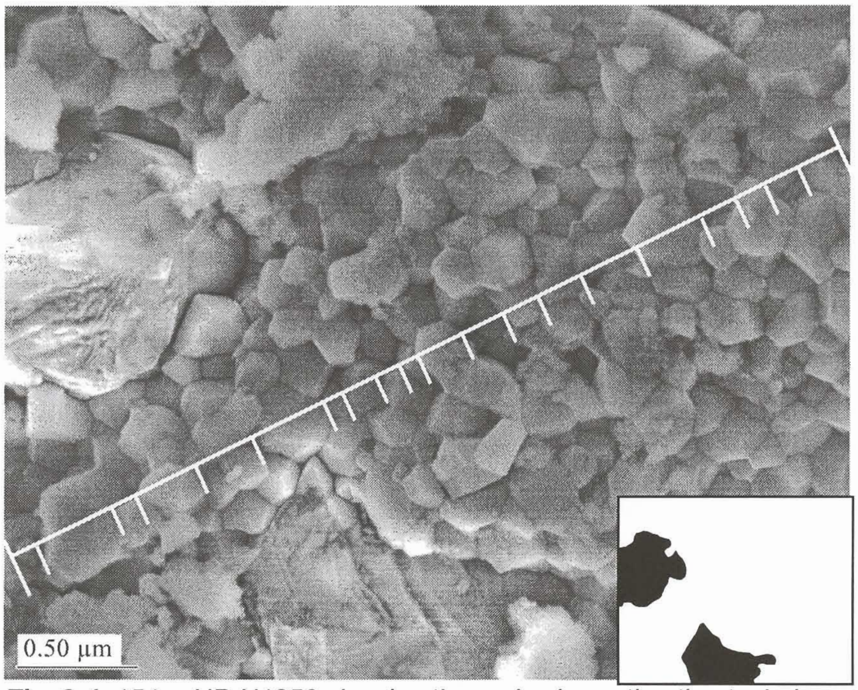


***Fig. 8-4**: 15◊-α-HP-H1250 showing the grain size estimation technique used.*

8.1.3 Equipment

All micrographs presented in this chapter were obtained with an ultrahigh resolution field emission SEM[‡] (scanning electron microscope) except fig. 8-1 and 8-2 which were obtained with a conventional (SEM)[†].

[‡] Jeol 6000, Laboratory for Microscopy and Microanalysis, Faculty of Science, University of Pretoria, South Africa.

[†] Jeol 840, as above.

8.2 Observations

Artificial fracture surfaces judged to be representative of each sample are given. For comparison, sets of micrographs are grouped on pages according the scheme given in table 8-2.

Average grain size as a function of HIPping temperature is given in fig. 8-24.

***Table 8-2**: Ordering of micrographs.*

Sample	Page	Micrographs	Remarks
0◊-α-Pα-H1400	60	Fig. 8-5	Both non-diamond containing
0◊-α-Pα-H1200		Fig. 8-6	
15◊-α-CP-H1400	61	Fig. 8-7	Same area
		Fig. 8-8	
		Fig. 8-9	
15◊-α-pH-H1300	62	Fig. 8-10	Same area
		Fig. 8-11	
		Fig. 8-12	
15◊-α-HP-H1250	63	Fig. 8-13	Same area
		Fig. 8-14	
		Fig. 8-15	
15◊-α-pH-H1250	64	Fig. 8-16	Same area
		Fig. 8-17	
		Fig. 8-18	
15◊-α-CP-H1250	65	Fig. 8-19	Same area
		Fig. 8-20	
		Fig. 8-21	
15◊-α-pH-H1300	66	Fig. 8-22	Close-up of diamond-alumina interface
		Fig. 8-23	

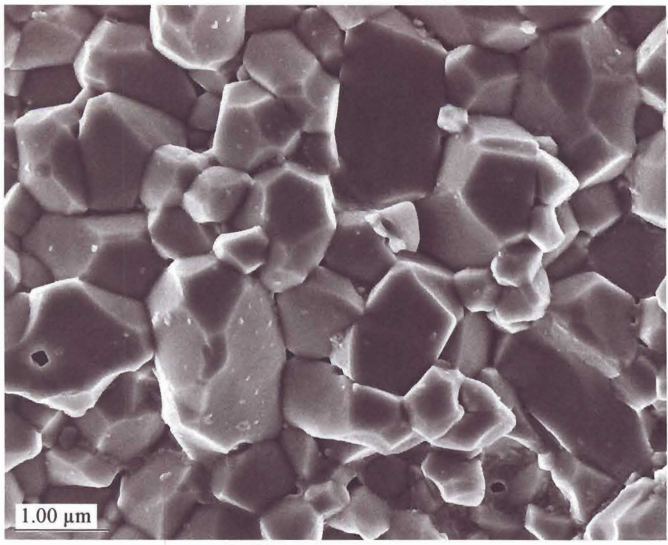


Fig. 8-5: *0◊-α-Pα-H1400.*

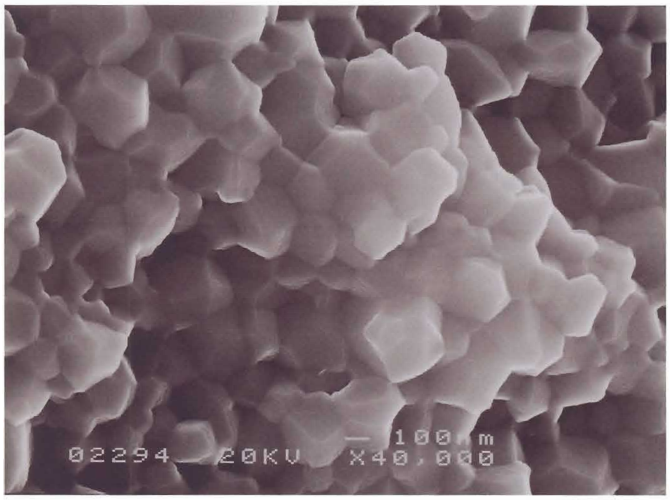


Fig. 8-6: *0◊-α-Pα-H1200.*

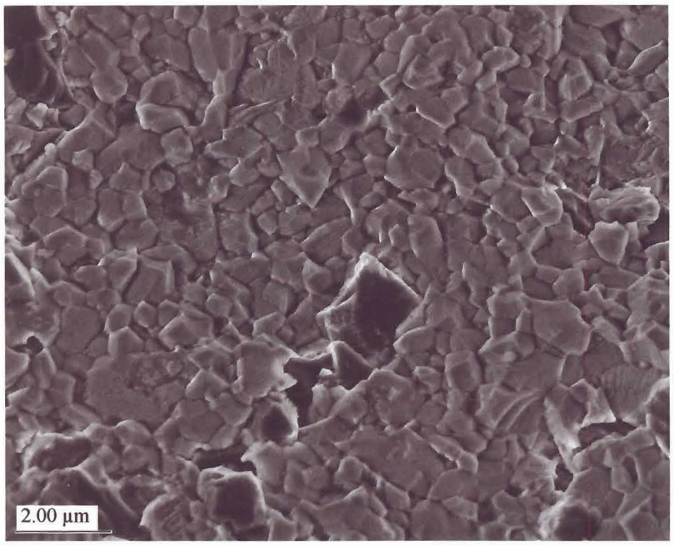
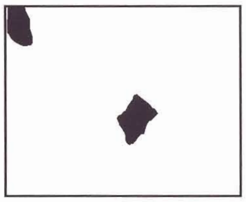


***Fig. 8-7**: 15∅-α-CP-H1400.*



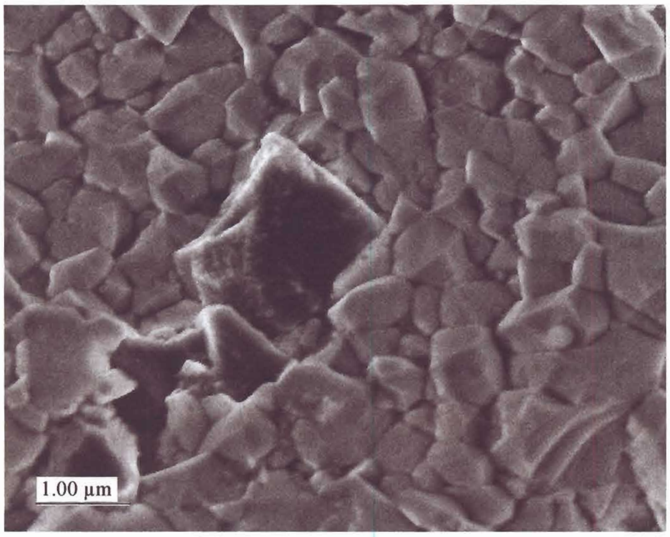
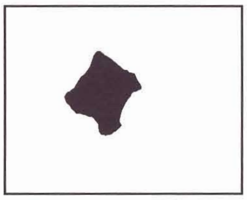


***Fig. 8-8**: 15∅-α-CP-H1400.*



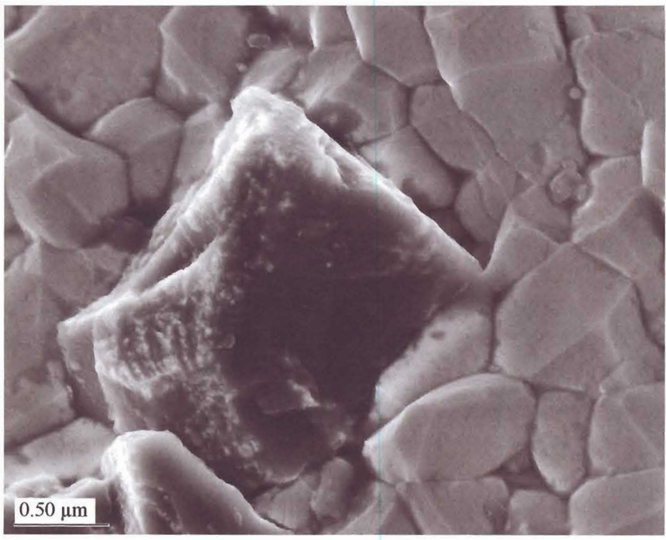
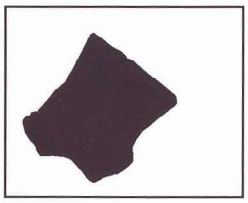


***Fig. 8-9**: 15∅-α-CP-H1400.*



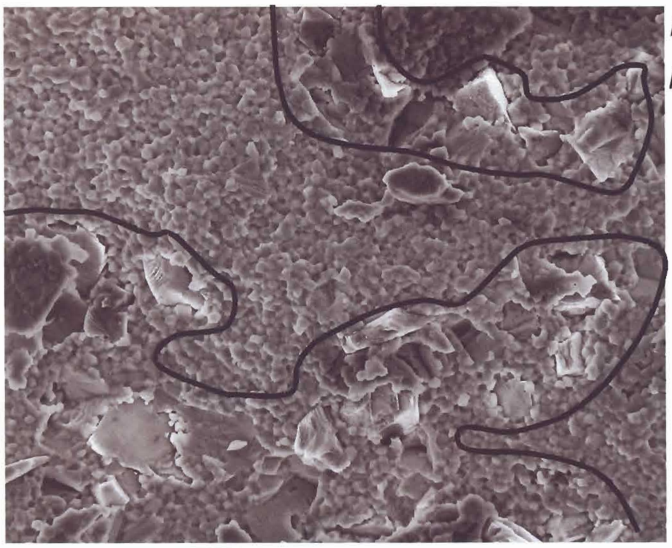
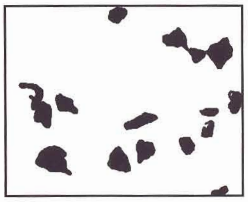


***Fig. 8-10**: 15◊-α-pH-H1300. Note grouping of diamond particles.*



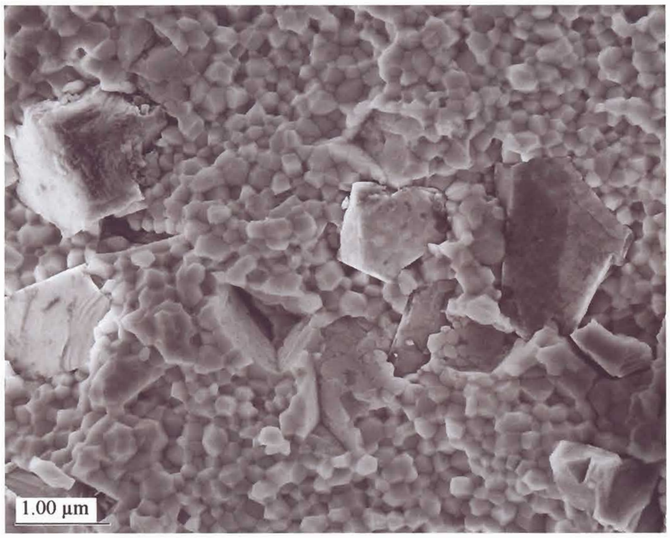
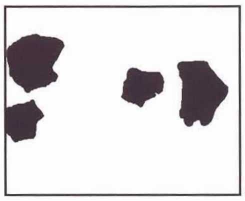


***Fig. 8-11**: 15◊-α-pH-H1300.*



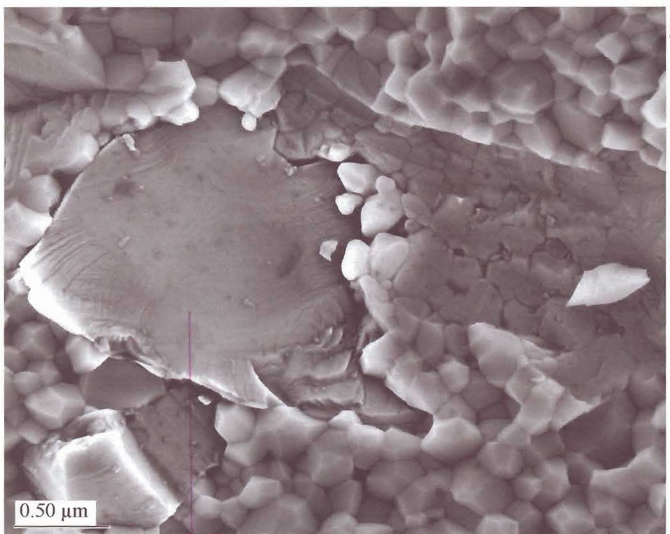
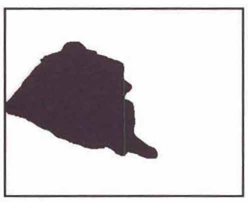
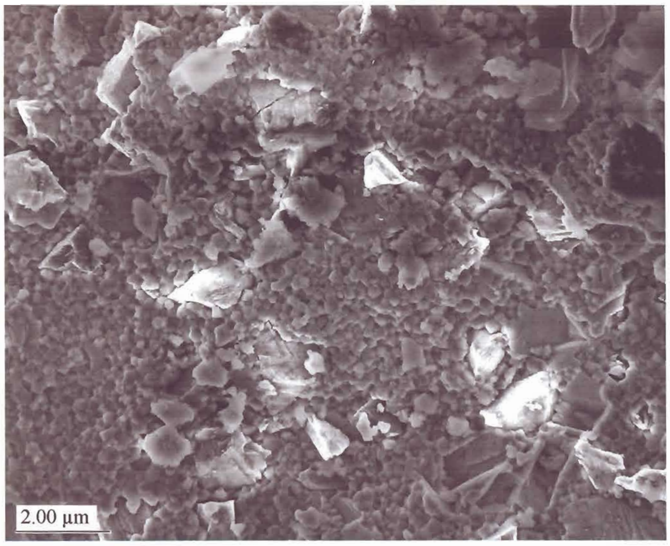


***Fig. 8-12**: 15◊-α-pH-H1300.*





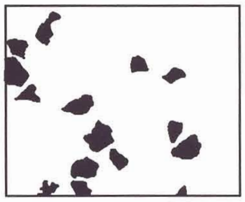
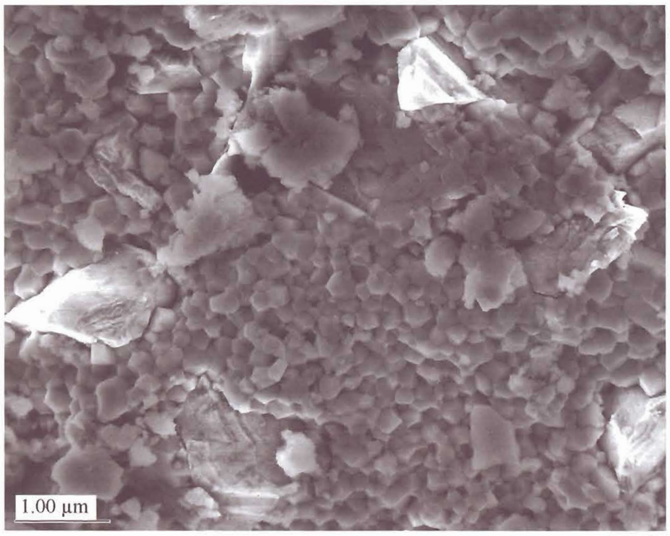


***Fig. 8-13**: 15ϑ-α-HP-H1250.*



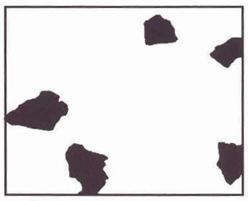
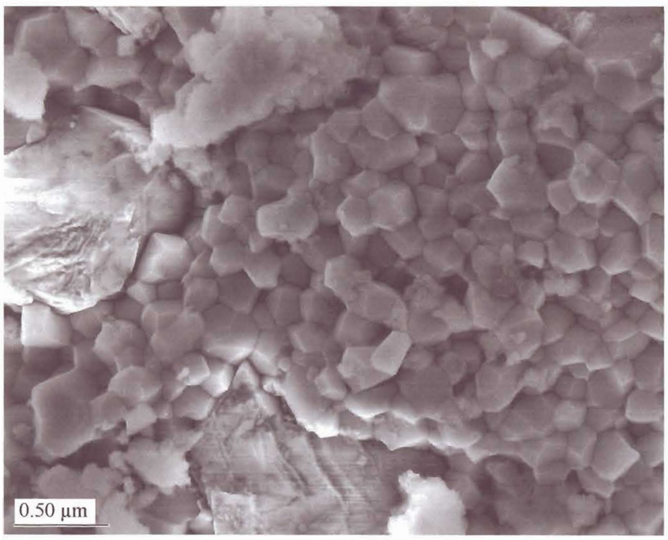


***Fig. 8-14**: 15ϑ-α-HP-H1250.*



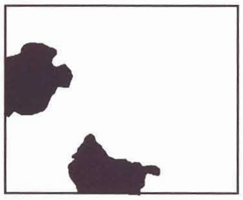
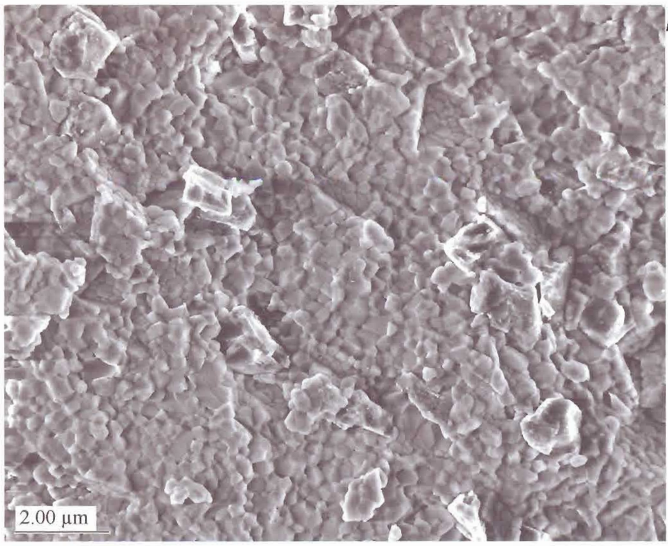


***Fig. 8-15**: 15ϑ-α-HP-H1250.*



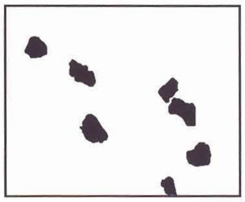
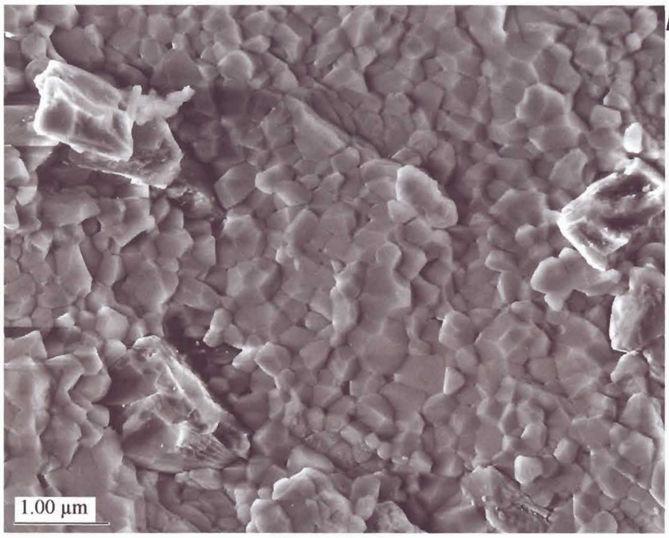


Fig. 8-16: 15φ-α-pH-H1250.



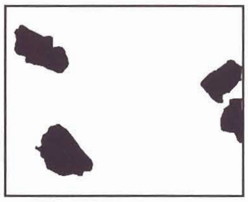
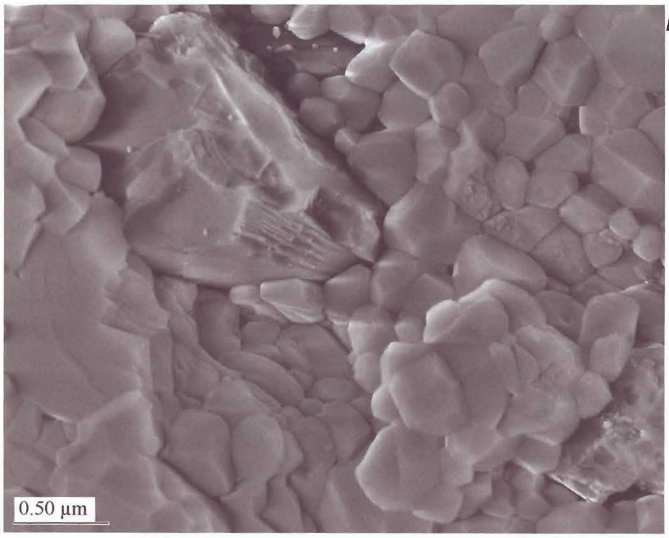


Fig. 8-17: 15φ-α-pH-H1250.



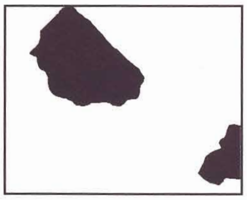


Fig. 8-18: 15φ-α-pH-H1250.

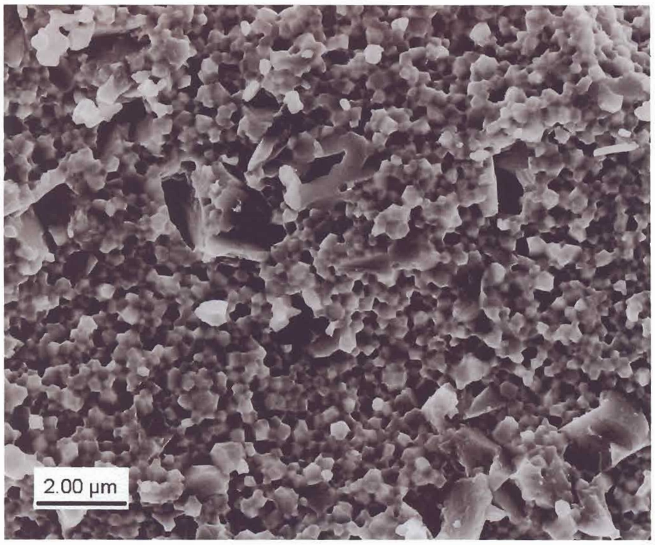
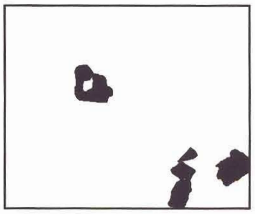


Fig. 8-19: *15◊-α-CP-H1250.*



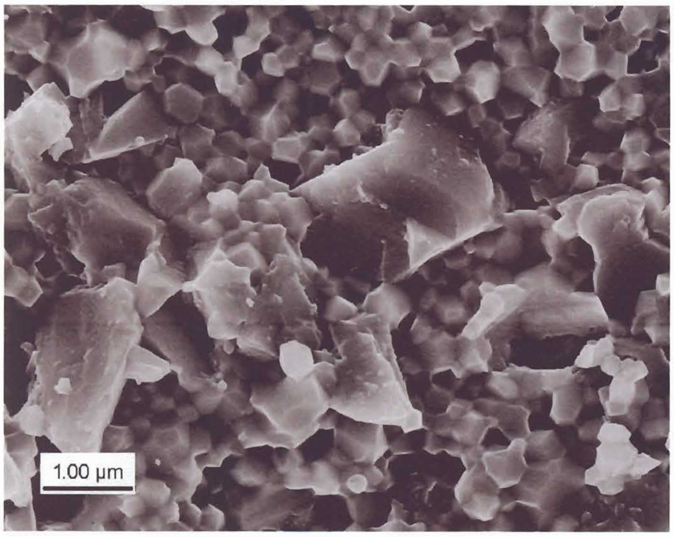
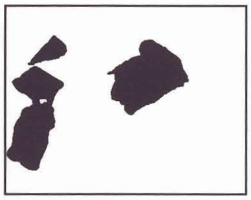


Fig. 8-20: *15◊-α-CP-H1250.*



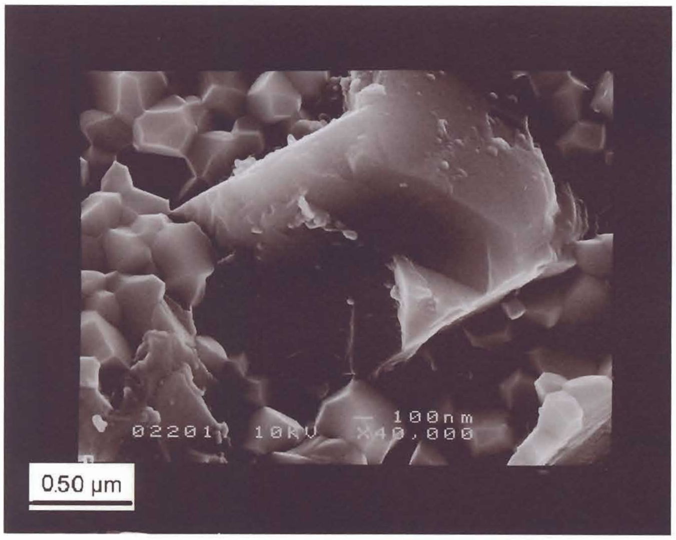
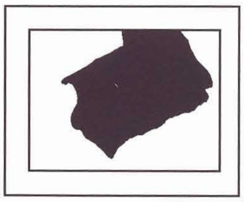


Fig. 8-21: *15◊-α-CP-H1250.*



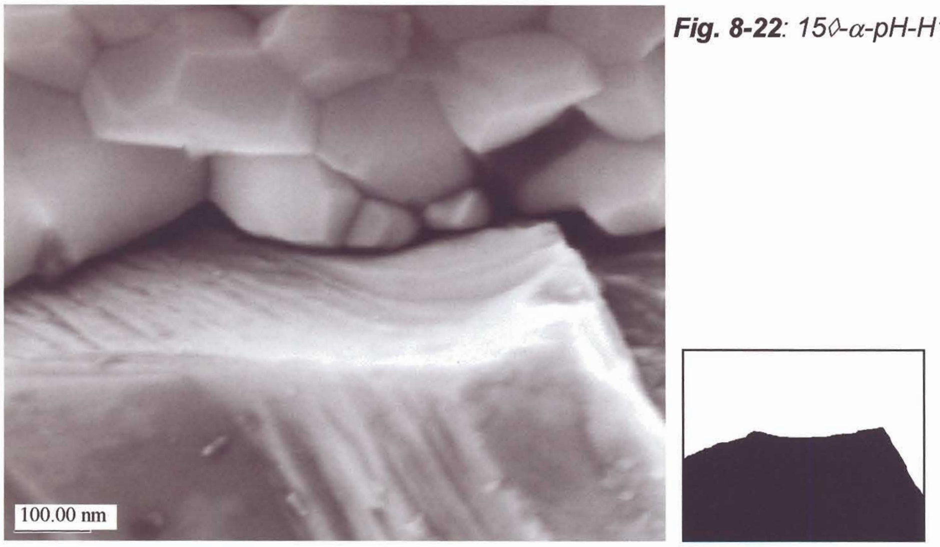


***Fig. 8-22**: 15◊-α-pH-H1300.*

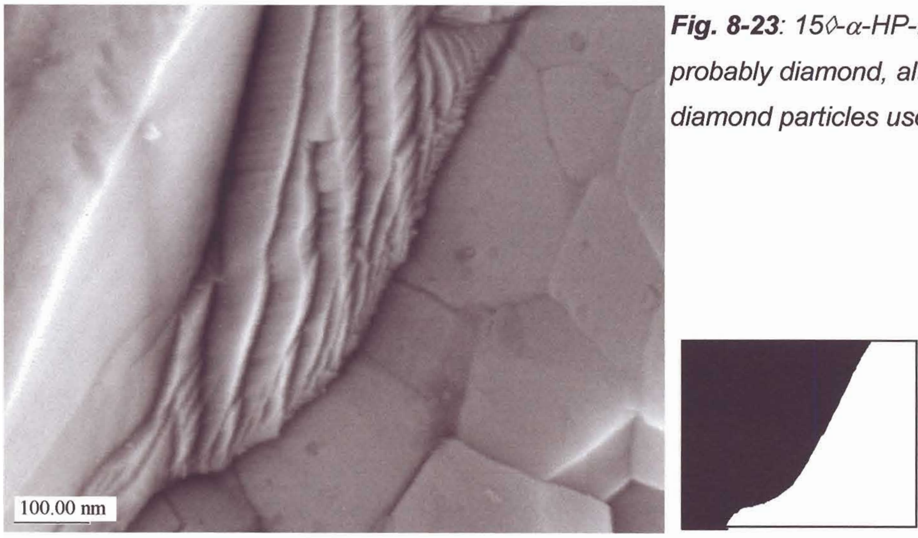


***Fig. 8-23**: 15◊-α-HP-H1300. The stepped surface is probably diamond, although this is not typical of the diamond particles used.*

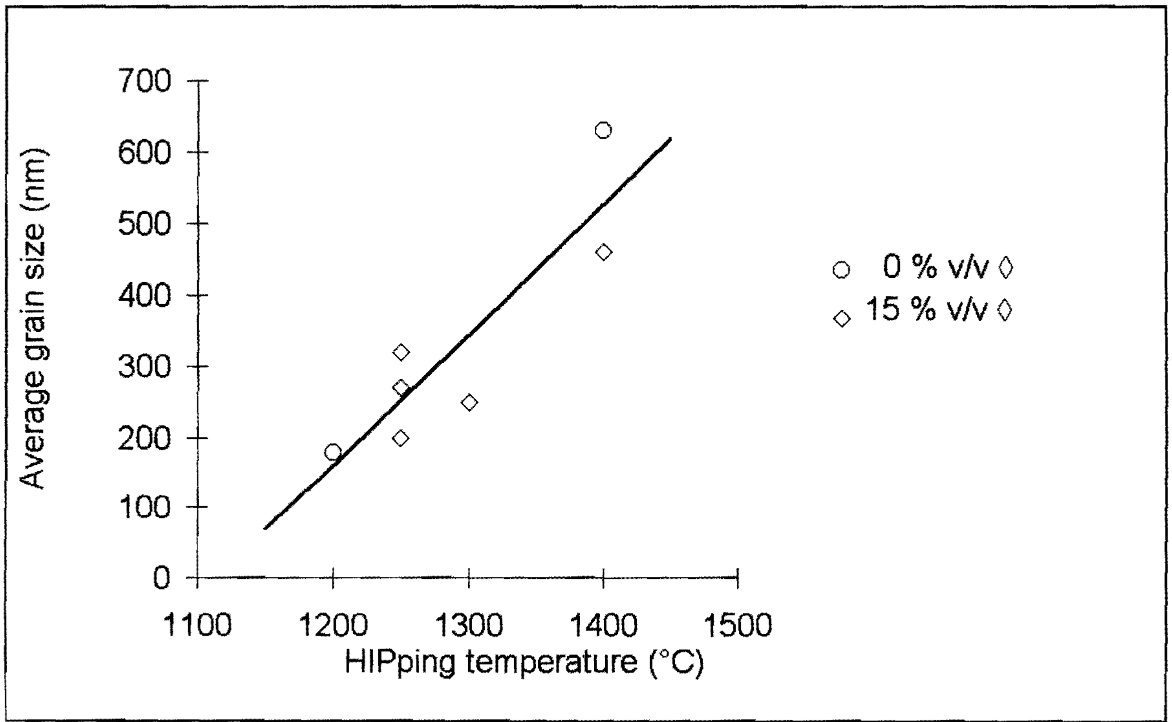


***Fig.8-24**: Average grain size, as defined in section 8-1, as a function of HIPping temperature.*