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**ANALYSIS OF THE SIZE AND
SHAPE OF ABRASIVES**

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ABSTRACT

A range of techniques has been used to evaluate the size and shape of a range of abrasive that have been used to carry out abrasive wear experiments on hardmetals and ceramics. Reasonable agreement was obtained between the results of image analysis size assessment and sieving experiments.

Little difference in shape parameters was seen for the abrasives examined. This was thought to be due to the fact that there was more difference in shape between on asperity and the next, and from particle to particle than there was between the abrasives that were examined.

The effectiveness of using confocal optical microscopy for the 3D analysis of the shape of abrasives was explored; good results were obtained.

Analysis of sand used in the ASTM B611 tests found that a significant fraction of the sand had been broken down in size by a factor of more than ten. The shape of the particles had become measurably less rounded

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CONTENTS

1	INTRODUCTION	1
2	EXPERIMENTAL METHOD.....	1
2.1	Materials and Microscopy	1
2.2	2D Image Analysis.....	2
2.3	Unwrapping Analysis	3
2.4	Sieving	4
2.5	Confocal Microscopy.....	5
3	RESULTS	6
3.1	Microscopy	6
3.2	Image Analysis	9
3.3	Confocal Microscopy.....	10
3.4	Sieving	13
3.5	Unwrapped Analysis.....	14
4	DISCUSSION AND CONCLUSIONS	17
5	ACKNOWLEDGEMENTS	17
6	REFERENCES.....	17

1 INTRODUCTION

The magnitude and mechanisms of abrasive wear are widely recognised to be heavily dependent on the size and shape of the abrasive particles that are causing the abrasion [1].

There is a long history of work that has investigated methods of analysing the shape of particles with the results of abrasion studies [2-11]. These studies have focused in the main on the analysis of the outline of sections through particles through simple shape analysis [1-3] Fourier analysis [4-5], analysis of the sharpness of asperities on the particles [6-10], and the curvature of the particle outline. Normally, optical and scanning electron microscopes are used to produce the images of particles. Only a small number of studies have attempted to analyse the 3-dimensional shape of particles, and these have relied on combining data from a number of 2D analyses.

To provide true 3D height information, other techniques are required. The height and lateral resolution and range of confocal optical microscopy are ideally suited to the analysis of abrasive particle shape. The resolution and range is controlled by the objective lens that is used, and in general an objective lens can be chosen that is suitable for most abrasive particles. Of course, optical microscopy is still governed by the resolution limits for light microscopy, so the technique is not applicable to abrasive particles with a size less than about 20 μm because of lateral resolution considerations.

2 EXPERIMENTAL METHOD

2.1 MATERIALS AND MICROSCOPY

Eight abrasive grits were examined. These were alumina and silica abrasives used in the ASTM B611 [12] and other abrasive tests. A range of four sizes of alumina were examined, three different sizes of a UK sourced silica abrasive, and the US Ottawa sand specified in the ASTM G65 [12] abrasive test.

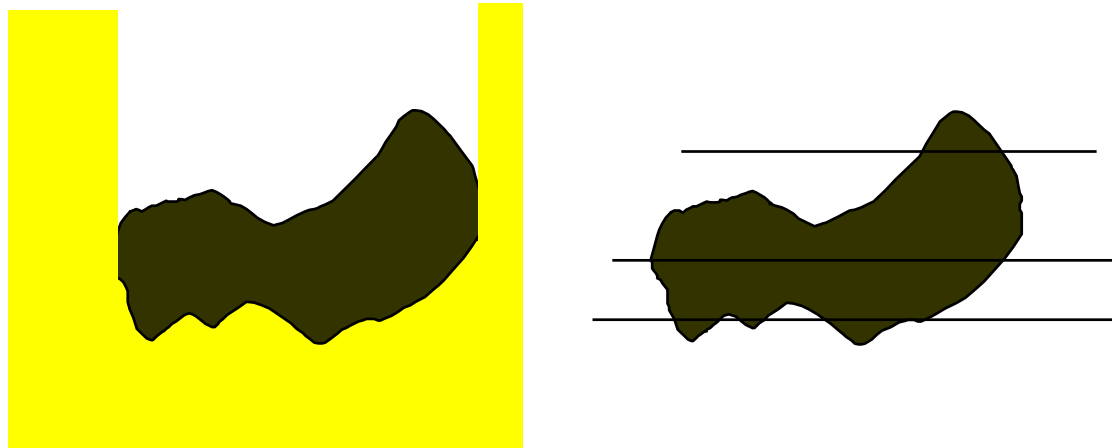
Measurement of the size and shape of abrasive particles was achieved both by 2D techniques, and also by direct 3D shape measurement through the application of the confocal scanning optical microscope [13].

With 2D imaging, two different approaches were adopted. The first was to mount some of the abrasive in epoxy, grinding and polishing until a cross section through the abrasive particles was produced. The epoxy mounting was a two-stage process with a small amount of epoxy mixed with the abrasive before the main epoxy was added. This was done to give some separation between the abrasive particles in the final cross section.

The second approach was to produce silhouette images of the abrasive particles. By scattering abrasive particles on a substrate and taking images. Ideally for image analysis good contrast is needed between the particles and the substrate background. This was found to be very difficult to achieve, particularly for the silica abrasives where the materials was translucent leading to light scattering within the grains so leading to unwanted artefacts in the images.

Attempts were made to produce high contrast images by SEM techniques, either by using a steel substrate to give good atomic number contrast, or by scattering the particles on a glass slide, coating with gold, and shaking off the particles to show shadows in the gold where the particles had been. Neither of these techniques was successful.

Eventually, it was decided to use manual analysis of the images by drawing around the individual particles to give particle outlines that could then be analysed. This procedure was facilitated by the loan of a tablet PC for 2 weeks. By displaying the image on the screen, the particles could be outlined in a very direct way.



Light Source

Figure 1 Schematic diagram showing two different methods to measure the size and shape of abrasive particles, a) forming silhouette outline by back illumination, b) sections through particles.

2.2 2D IMAGE ANALYSIS

Several different methods were used to analyse the images obtained. Figure 2 shows a schematic diagram describing some simple measures of the particle size, the maximum diameter, the minimum diameter and the equivalent circle diameter (the diameter of a circle with the same area as the area of the particle). The area and perimeter of each particle were also determined.

Simple shape factors that were then determined. These were the circularity (defined as $4A\pi/P^2$) where A is the projected particle area and P is the projected particle perimeter), and the ratio of the maximum and minimum diameters of the particles. Both the ratio and the circularity are factors that are 1 for a circle and decrease as the angularity of the particle decreases. About 70 particles were measured for each abrasive.

The SPQ parameter (defined in detail later) was also calculated. This parameter provides a measure of the average shape of the asperities on the particles that are most likely to cause wear damage. These are those asperities that protrude most from the particle.

A procedure to calculate the SPQ parameter was written as a macro for the Image J system (Figure 3). This chooses the asperities that are larger than equivalent circle, finds the extreme of the asperity, and fits quadratic curves to the sectors from the apex to the equivalent circle. The angle at the apex between the two quadratics is then determined for every asperity. The SPQ parameter is then the average of the cosine of half of the apex angle taken over all of the asperities in the sample. In contrast to the simple shape parameters described earlier, the SPQ parameter should be zero for round particles and becomes closer to 1 the sharper the asperity.

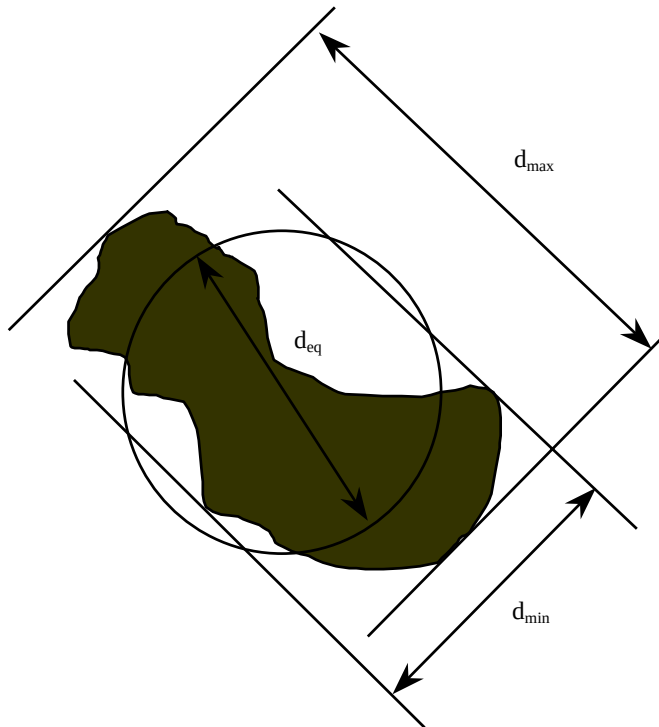


Figure 2 Schematic diagram showing basic diameters measured in image analysis.

2.3 UNWRAPPING ANALYSIS

Another approach that was used was to make use of the alternative description of the particle that is obtained by “unwrapping” the shape of the particle. Figure 4 explains the principle. The centroid of the particle is found and the perimeter (r, θ) values used to form a data set that completely describes the outline of the particle. The advantage of this process is that the unwrapped profile can be treated with many different signal processing algorithms that have been developed over many years such as fast Fourier transform analysis (FFT). A macro was written for the Image J system to extract the unwrapped perimeter data sets, and a programme written in Labview was used to analyse the unwrapped data sets.

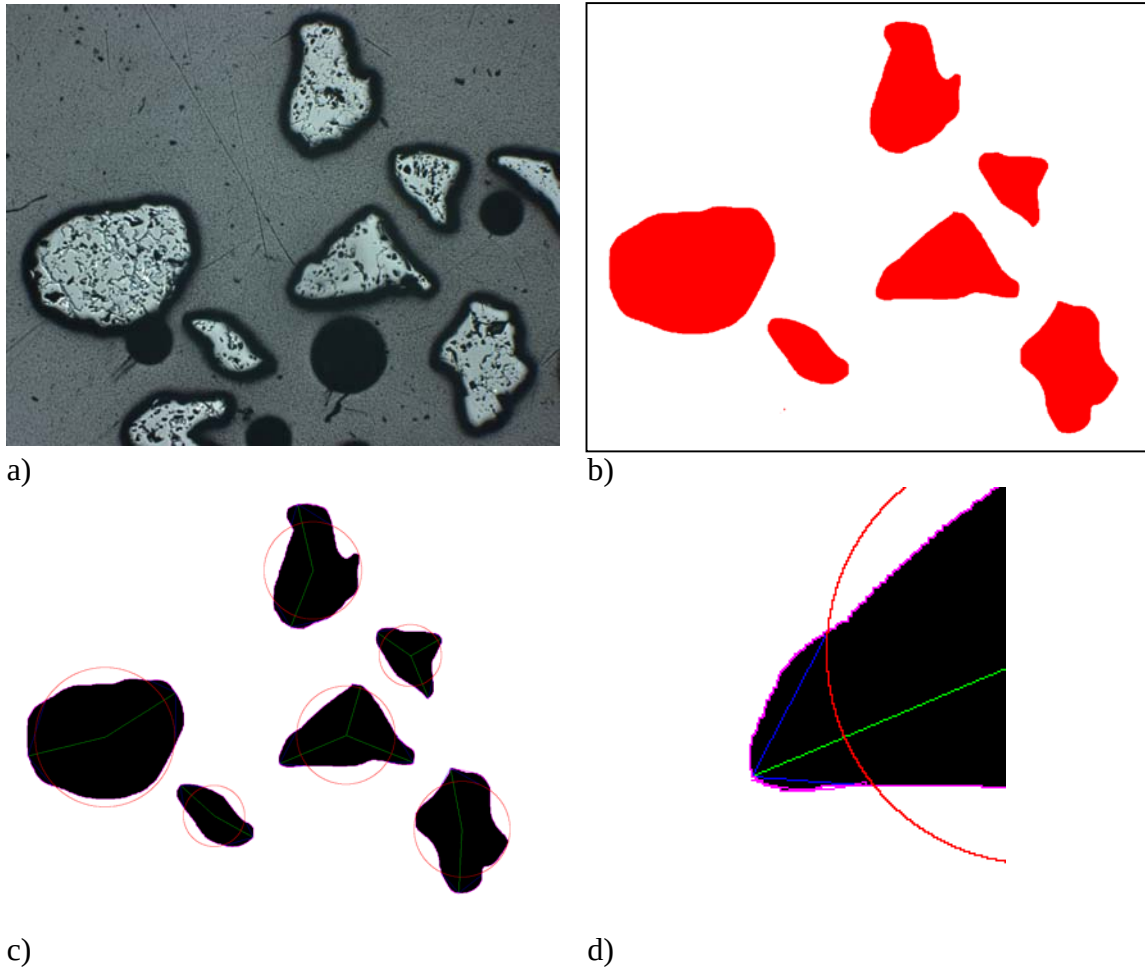
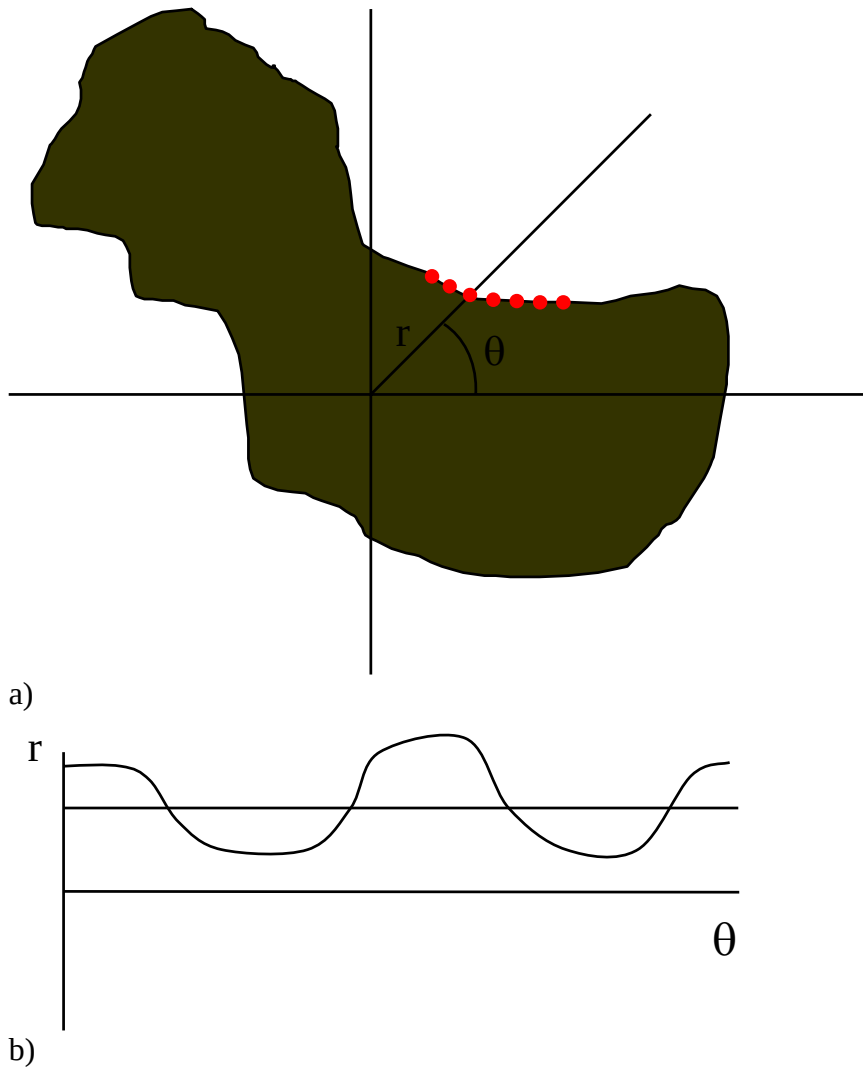


Figure 3 Determination of SPQ parameter, a) original image, b) thresholded binary image, c) SPQ analysis; red is equivalent circles, green - radii to apex of asperities, purple – quadratic fits to sectors at asperity, d) enlarged view of single asperity.

2.4 SIEVING

To complement the measurement of size and shape through image analysis, traditional sieving of the particles through a sequence of sieves was used to determine the size of the abrasive grits. The sequence of sieves used was 710, 600, 500, 425, 355, 300, 250, 212, 180, 150, 125, 106, 90, 75, 50 μm . The weight of abrasive retained at each sieve was measured and was used to calculate the percentage of abrasive that passed through that sieve. About 100 gm of abrasive was used of each abrasive.



b)
Figure 4 Forming “unwrapped” profile for particle, a) schematic of particle and unwrapping process, b) resultant profile.

2.5 CONFOCAL MICROSCOPY

The 25/36 silica and the F30 abrasives were examined by confocal microscopy. This instrument has the ability to form “optical slices” that contain image information from just the focal plane of the microscope (Figure 5). When the height of the sample is scanned, these optical slices can be combined to form a three dimensional height map, and a corresponding simple image of the object being examined.

The height maps can be analysed to extract true 3D shape information for asperities on the particles.

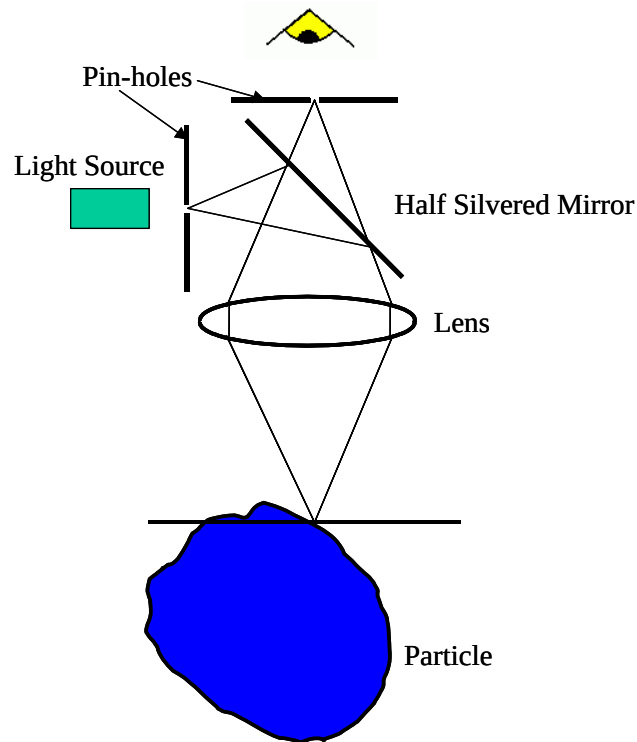
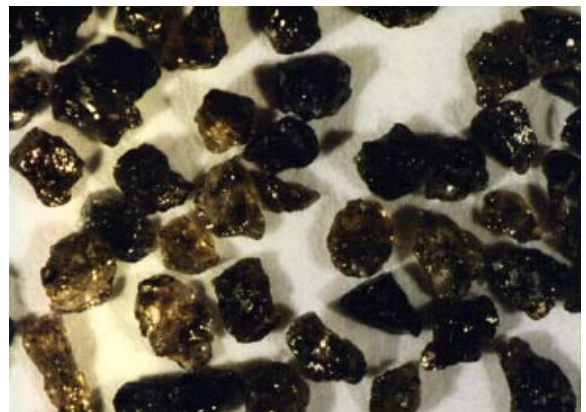
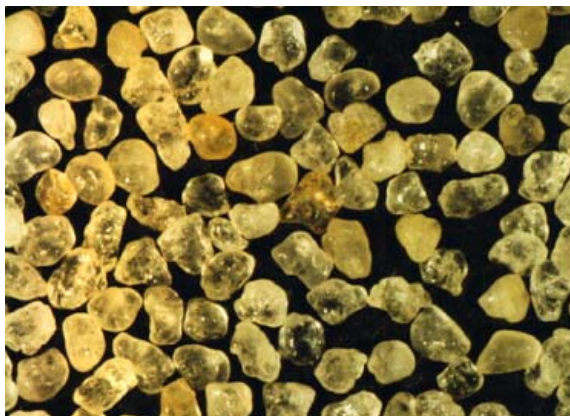


Figure 5 Schematic diagram of the imaging process for a confocal microscope

3 RESULTS

3.1 MICROSCOPY

Figure 6 shows typical macroscopic images of the two large abrasives. The large sand is light in colour and is obviously translucent, whereas the alumina is dark and opaque. Figure 7 shows typical silhouette images of all the particles examined, and Figure 8 shows images of polished cross sections made through the particles.



a) b)
Figure 6 Optical micrographs of abrasives. Whole particles. Images optimised for conventional microscopy using contrasting backgrounds, a) 25-30 silica, b) F30 alumina

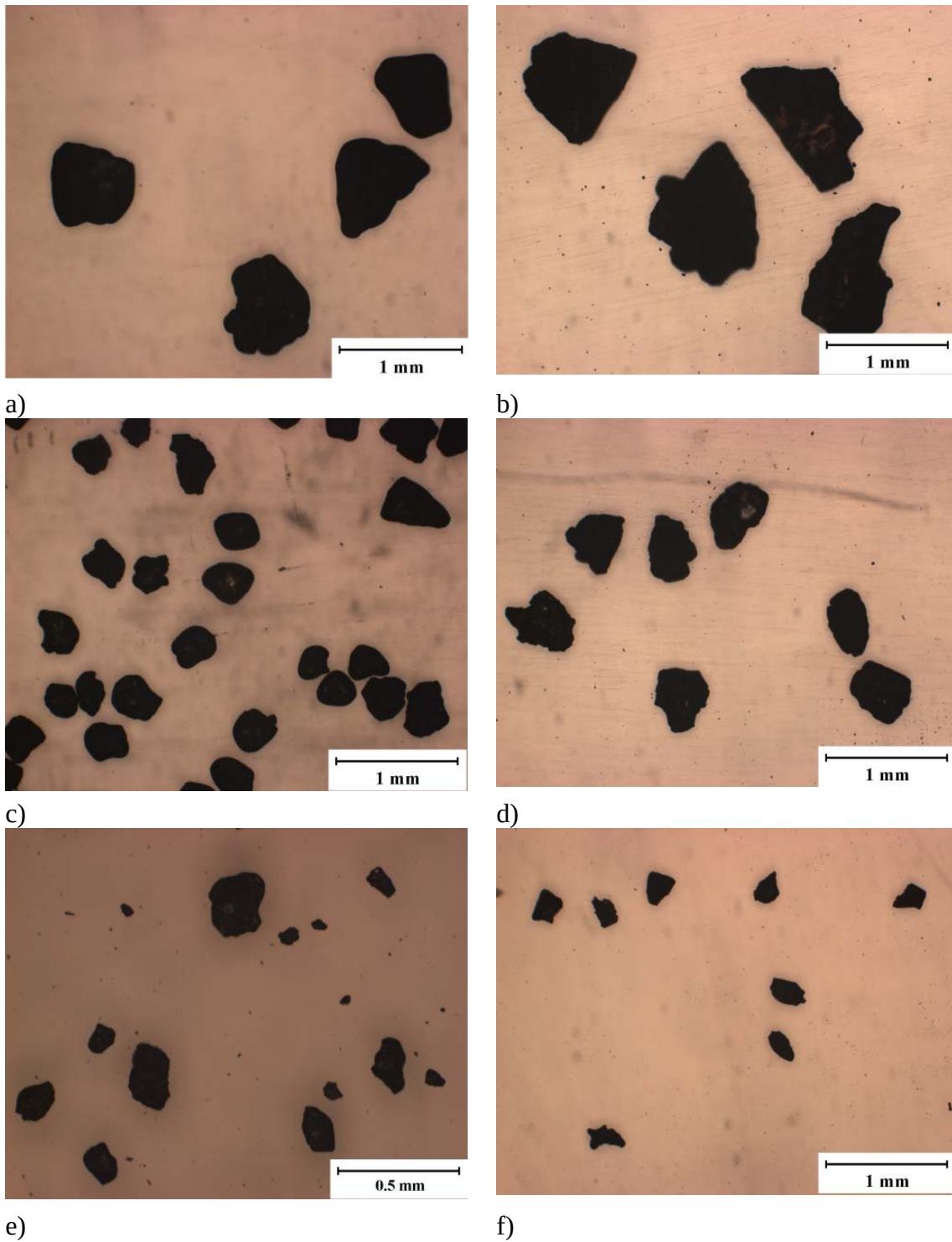


Figure 7, Optical micrographs of abrasives. Whole particles. Micrographs are optimised to obtain outlines of the particles. a) 25-30 silica, b) F30 alumina, c) 50-70 sand, d) F60 alumina, e) fine sand, f) F100 alumina.

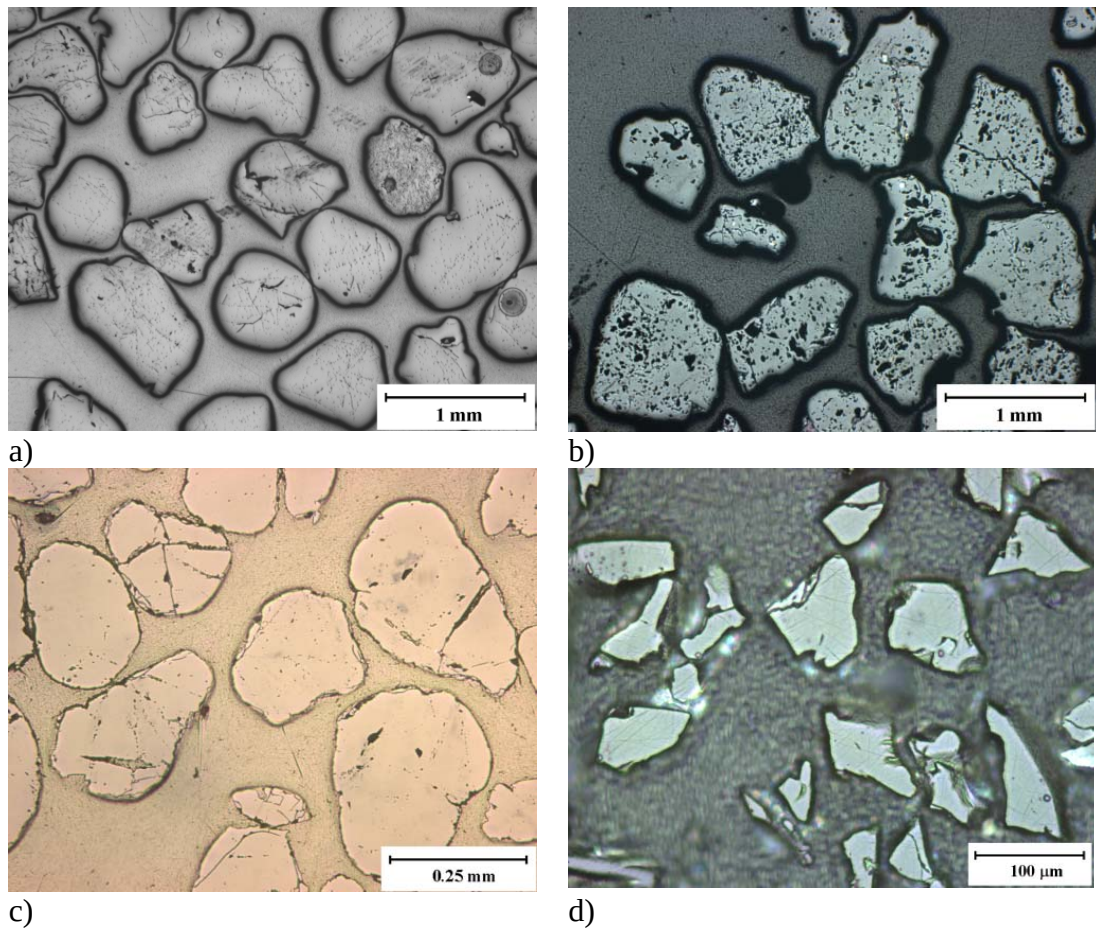


Figure 8 Optical micrographs of abrasives. Sections through particles. a) 25-30 silica, b) F30 alumina, c) 50-70 sand, d) F100 alumina

The polished cross section images show some evidence for fracture in the single normally crystal sand particles. By contrast, the large alumina particles are polycrystalline, with a small proportion of second phase present.

Figure 9 shows images of the 25-36 sand and the F30 alumina abrasive both unused and after it has been used in the ASTM B611 test to abrade WC/Co hardmetals. For the alumina, there is little visible sign of change of size or shape in the abrasive from before the test to after the test. With the 25-36 sand there are two size populations of sand visible. The larger particles are similar in size to the untested abrasive, but there is also a substantial fraction of much finer material present.

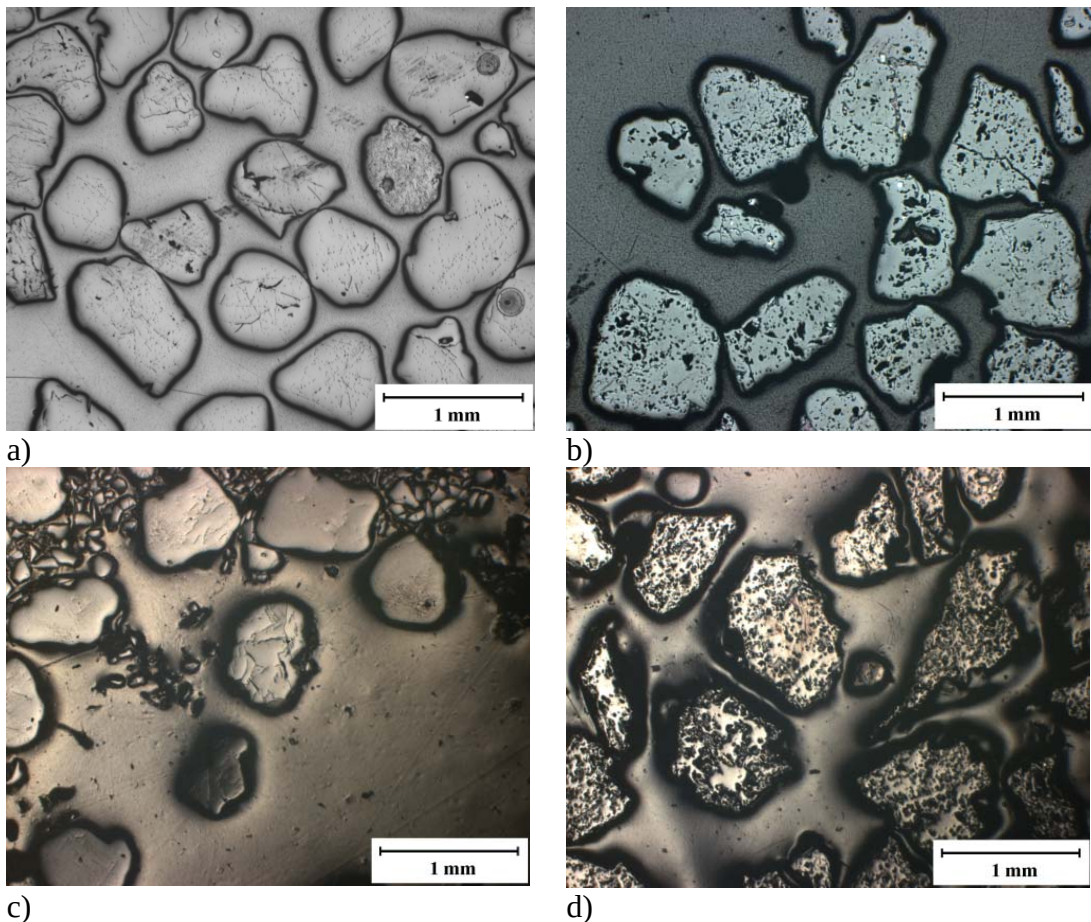


Figure 9 Optical micrographs of abrasives. Sections through particles. a) fresh 25-30 silica, b) fresh F30 alumina, c) 25-36 silica after one use in ASTM B611 test, d) F30 alumina after one use in ASTM B611 test

3.2 IMAGE ANALYSIS

The numerical results of the image analysis are given in Table 1. Results for both the 3D silhouette and the section measurements are given. As expected, the 3D size of the particles is always larger than the section size. The circularity and the ratio parameters are very similar for all the abrasives measured, and there seems to be a small increase in these parameters for the sand compared with the closest equivalent alumina abrasives (ie the sand is slightly rounder than the alumina). However, these differences are always within one standard deviation of the mean values, so the significance of this observation is quite weak.

The SPQ parameter seems to show a slight decrease for the sand compared to the alumina, again suggesting that the sand is slightly rounder than the alumina, but the difference is also of the same order as the size of the calculated standard deviations.

The sand specified for abrasion testing within the ASTM G65 standard is almost identical in size and shape to the alternative UK sourced 50-70 sand.

Table 1, Measured parameters for size and shape for alumina and sand abrasives. Please note that size parameter in the table is the equivalent circle diameter.

	Size, μm		Circularity		Ratio		SPQ	
	3D	Section	3D	Section	3D	Section	3D	Section
F30 Alumina	879 $\pm$ 115	645 $\pm$ 170	0.70 $\pm$ 0.06	0.72 $\pm$ 0.10	0.72 $\pm$ 0.13	0.68 $\pm$ 0.15	0.66 $\pm$ 0.19	0.70 $\pm$ 0.18
F60 Alumina	419 $\pm$ 50	366 $\pm$ 54	0.70 $\pm$ 0.08	0.75 $\pm$ 0.06	0.66 $\pm$ 0.15	0.74 $\pm$ 0.13	0.69 $\pm$ 0.17	0.67 $\pm$ 0.18
F100 Alumina	209 $\pm$ 24		0.68 $\pm$ 0.10		0.66 $\pm$ 0.16		0.71 $\pm$ 0.20	
F320 Alumina	41 $\pm$ 11	22 $\pm$ 9	0.72 $\pm$ 0.10	0.57 $\pm$ 0.12	0.64 $\pm$ 0.17	0.55 $\pm$ 0.17	0.72 $\pm$ 0.19	0.78 $\pm$ 0.20
25-36 Sand	665 $\pm$ 49	553 $\pm$ 114	0.78 $\pm$ 0.05	0.72 $\pm$ 0.09	0.75 $\pm$ 0.12	0.70 $\pm$ 0.14	0.61 $\pm$ 0.18	0.68 $\pm$ 0.18
50-70 Sand	294 $\pm$ 43	167 $\pm$ 60	0.79 $\pm$ 0.05	0.71 $\pm$ 0.10	0.77 $\pm$ 0.10	0.69 $\pm$ 0.14	0.63 $\pm$ 0.19	0.70 $\pm$ 0.20
Fine Sand	106 $\pm$ 41		0.76 $\pm$ 0.06		0.72 $\pm$ 0.13		0.68 $\pm$ 0.19	
Ottawa Sand	344 $\pm$ 30	200 $\pm$ 59	0.80 $\pm$ 0.03	0.68 $\pm$ 0.11	0.77 $\pm$ 0.11	0.64 $\pm$ 0.14	0.64 $\pm$ 0.17	0.74 $\pm$ 0.18

Table 2 shows a comparison in the measured size and shape parameters between the abrasive that was passed through the ASTM B611 test and unused abrasive. There is no significant difference between the alumina comparing the unused and used abrasive (the perceived increase in size is well within one standard deviation of the data). For the sand, although the size and shape of the large particles had not altered significantly for the large fraction of particles, the size of the small particles had been reduced by a factor of ten to 48 μm . The circularity and ratio shape factors were also significantly smaller, with the SPQ parameter significantly larger. All three of these shape factors suggest that the small fragmented sand particles are significantly less rounded than the original sand.

Table 2, Measured parameters for size and shape of alumina and sand abrasives both before and after use in ASTM B611 test. Note that there are two columns for the used 25-36 sand corresponding to the large and small sand fractions respectively.

	F30 Alumina	F30 Used	25-36 Sand	25-36 Sand Used, Large Fraction	25-36 Sand Used, Small Fraction
Size	645 $\pm$ 170	698 $\pm$ 252	553 $\pm$ 114	595 $\pm$ 150	48 $\pm$ 28
Circularity	0.72 $\pm$ 0.10	0.73 $\pm$ 0.10	0.72 $\pm$ 0.09	0.79 $\pm$ 0.05	0.59 $\pm$ 0.12
Ratio	0.68 $\pm$ 0.15	0.72 $\pm$ 0.14	0.70 $\pm$ 0.14	0.74 $\pm$ 0.12	0.51 $\pm$ 0.19
SPQ	0.70 $\pm$ 0.18	0.68 $\pm$ 0.18	0.68 $\pm$ 0.18	0.65 $\pm$ 0.18	0.80 $\pm$ 0.19

3.3 CONFOCAL MICROSCOPY

Typical particle images and their corresponding height maps obtained by confocal microscopy are shown in Figure 10. Figures 10a and 10b are in-focus images and are difficult to interpret by comparison with conventional optical images because of the lack of depth information in the images. Surface reconstructions of typical particles are shown in Figures 10e and 10f for the two different powders.

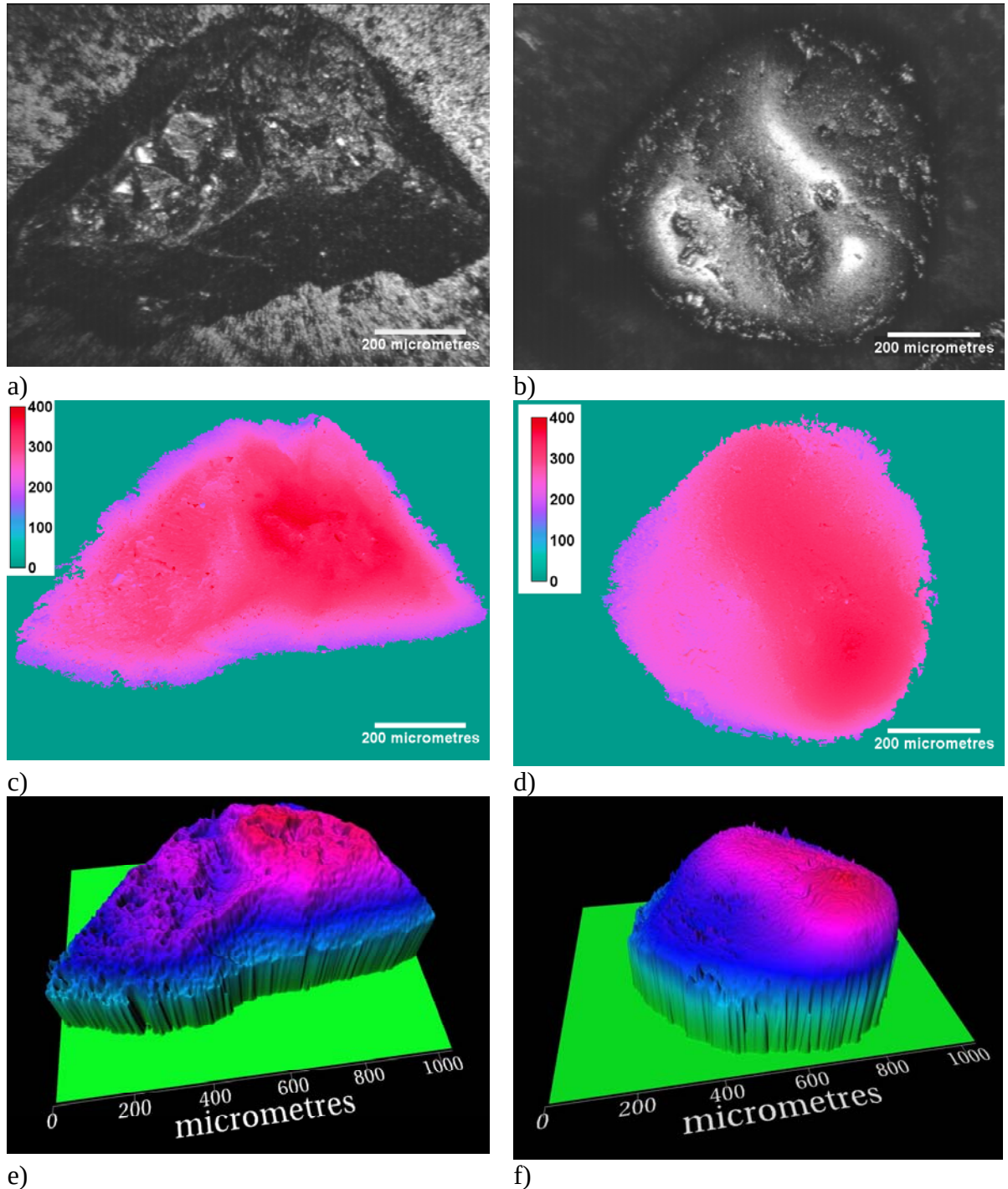
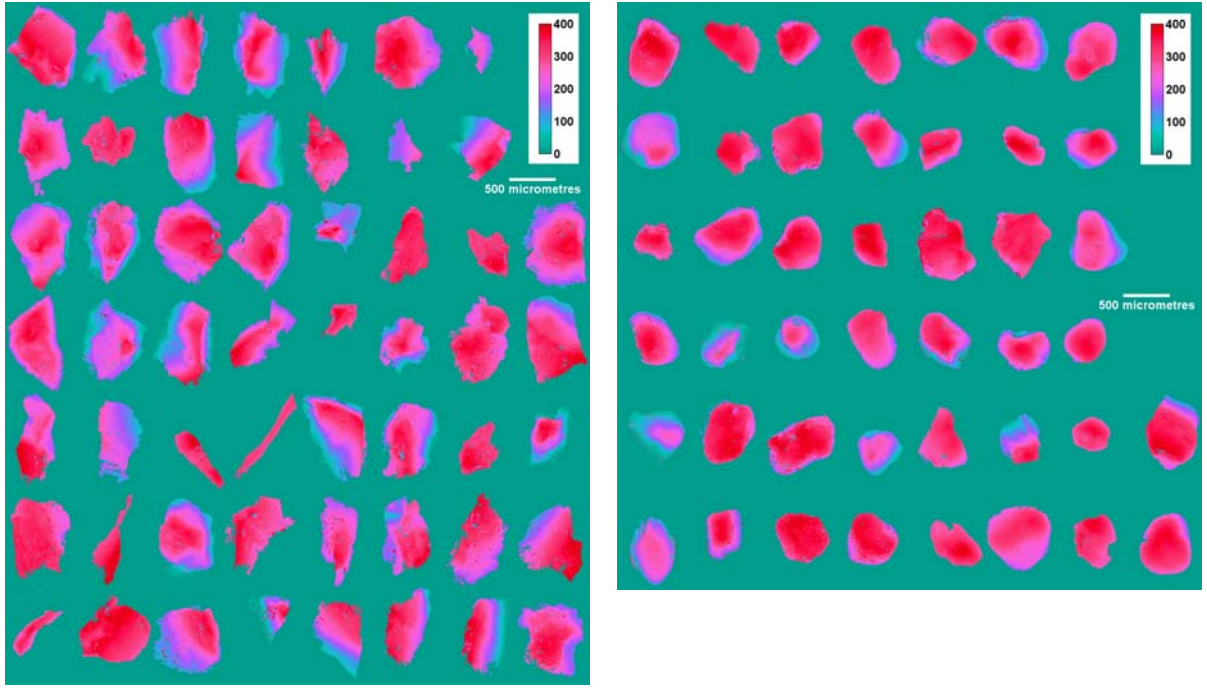


Figure 10, Results of confocal optical microscope measurements on single particles, a) image of alumina particle, b) image of silica particle, c) height map for particle in a) (height scale in micrometres), d) height map for particle in b) (height scale in micrometres), e) surface reconstruction for particle in a), f) surface reconstruction for particle in b).

Montages of height maps for the particles analysed in this study are shown in Figure 4 to give an impression of the variability within one abrasive type and to allow a qualitative comparison between both abrasive types. Particles for which there was too much noise were rejected although an attempt was made to reduce any bias in the particles data sets discarded during this process. Inspection of the montages confirms that the alumina particles appear to be more angular than the silica particles.



a) b)
Figure 11, Montages of all particles analysed in this study, a) alumina particles, b) silica particles.

To calculate the particle cross-section area versus depth (measured from the highest part of the particle), a histogram was created from the height map images after processing. The cross-sectional area was calculated by summing the histogram bins. The results are shown in Figures 12a and 12b.

Obviously the particles are different shapes and sizes. Therefore to perform a preliminary analysis it was helpful to normalise the area versus depth curves. This could be done by calculating the maximum area and the depth of the maximum area and then using these parameters to normalise the depth and area. But the maximum area can only be found for those area versus depth curves that exhibit a horizontal portion. To enable normalisation to be carried out on all the curves, the particles were approximated as ellipsoids;

For an ellipsoid defined by

$$\frac{x^2}{a^2} + \frac{y^2}{b^2} + \frac{z^2}{c^2} = 1$$

Calculating the depth, d , along the y axis starting from $y=b$ ($d=0$) gives an area A

$$A = \frac{\pi ac(2bd - d^2)}{b^2}$$

Normalising A by πac , the maximum area and d by b , the maximum depth gives

$$q = 2p - p^2$$

where $q = A/\pi ac$ and $p=d/b$

This allowed a multivariate linear regression to be made using $2p$ and $-p^2$ as the two terms and hence estimates for b and πac to be made for each curve.

This removed some of the size variation of the particles and allowed the shape variation to be more easily studied and can be seen in Figures 11c and 11d.

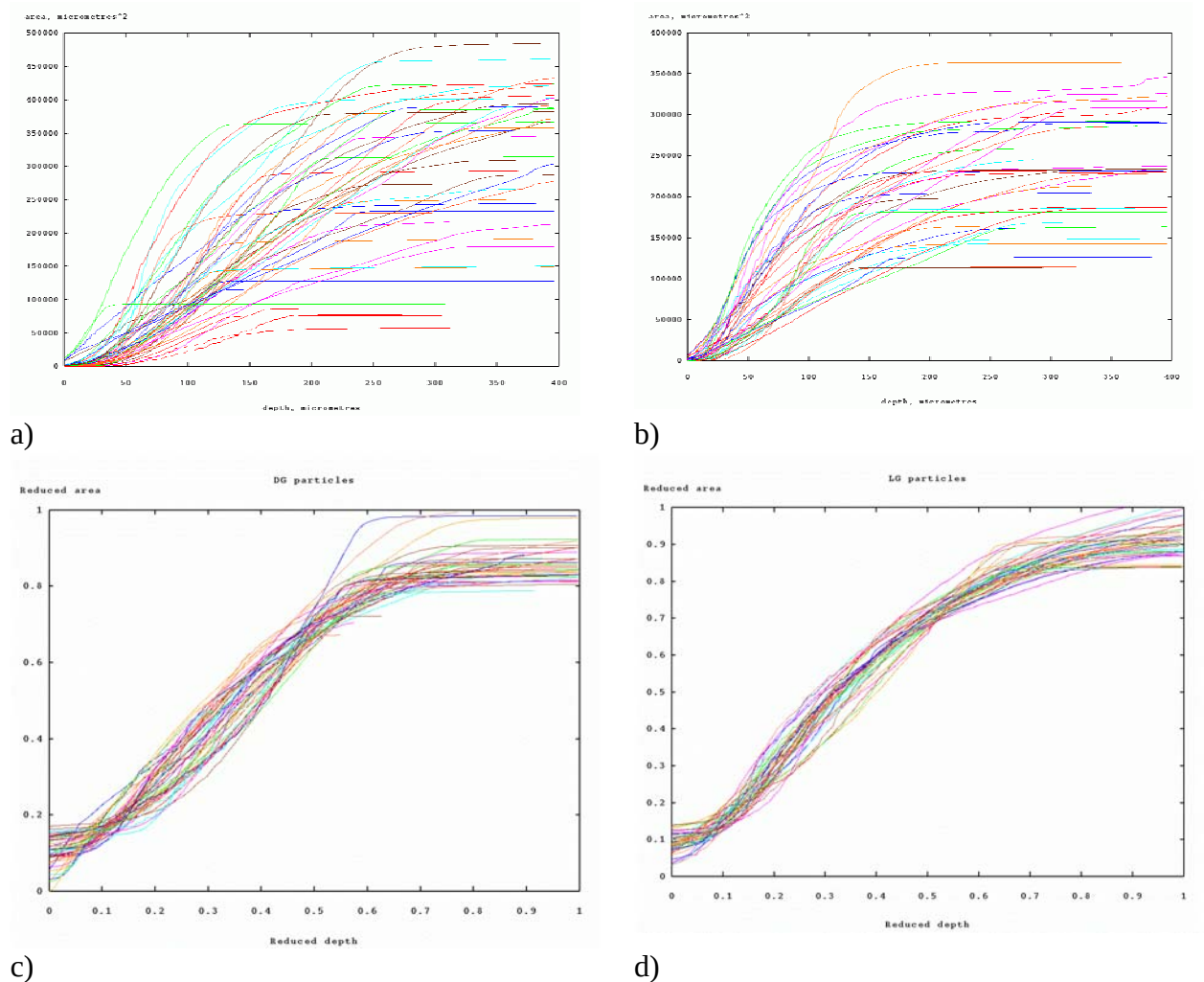


Figure 11, Area-depth curves for particles, a) alumina, b) silica, c) normalised alumina, d) normalised silica

3.4 SIEVING

The results of the sieving are shown in Figure 12. They are broadly in line with the results of the image analysis shown earlier.

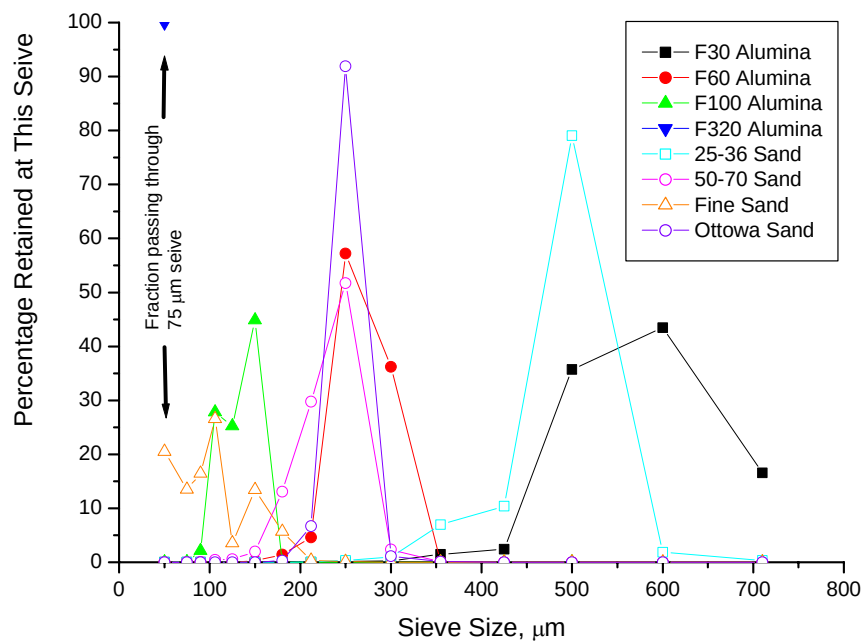


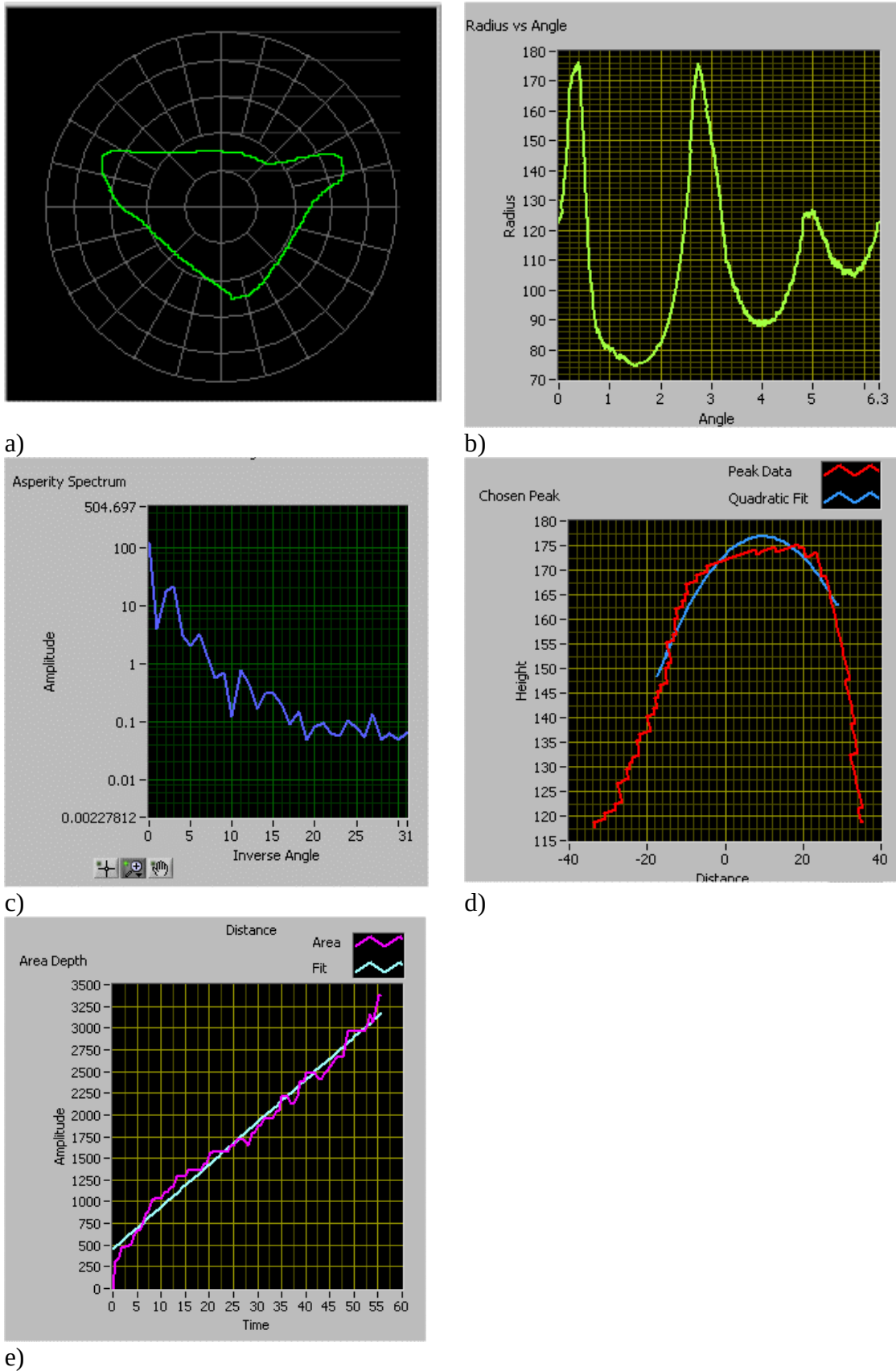
Figure 12, Results of sieving on the abrasives examined.

3.5 UNWRAPPED ANALYSIS

The (r, θ) data sets of the particles that were produced by the unwrapping process were analysed in a number of ways (Figure 13). These were:

- Fourier analysis of the (r, θ) datasets and determination of the slope of the initial four peaks with respect to inverse angle (F-ratio). For a round particle the Fourier analysis will yield a delta function, so the F-ratio will increase very quickly as the particle becomes closer and closer to a circle. It should be noted that this parameter applies directly to the full particle.
- Fitting a parabolic curve to specific asperities and determining the curvature of the parabola at the apex. This is equivalent to determining the second order coefficient c of the fitting parabola $a + bx + cx^2$. This parameter has to be determined individually for all asperities.
- Determining the area-depth characteristic of asperities and find the slope of this curve. This has to be carried out on a single asperity basis.

Figure 13a shows the original particle outline that is unwrapped to form the profile in Figure 13b. In many ways this process itself is a good way of comparing the particle outline to a circle as a circle would give a horizontal line on this Figure. The deviation from this horizontal line gives a measure of the deviation of the particle from a round particle.



e) Figure 13, Particle analysis by particle unwrapping, a) original particle outline, b) unwrapped profile, c) FFT of unwrapped profile, d) fit of parabola to first asperity in b), e) area-depth curve for asperity in d)

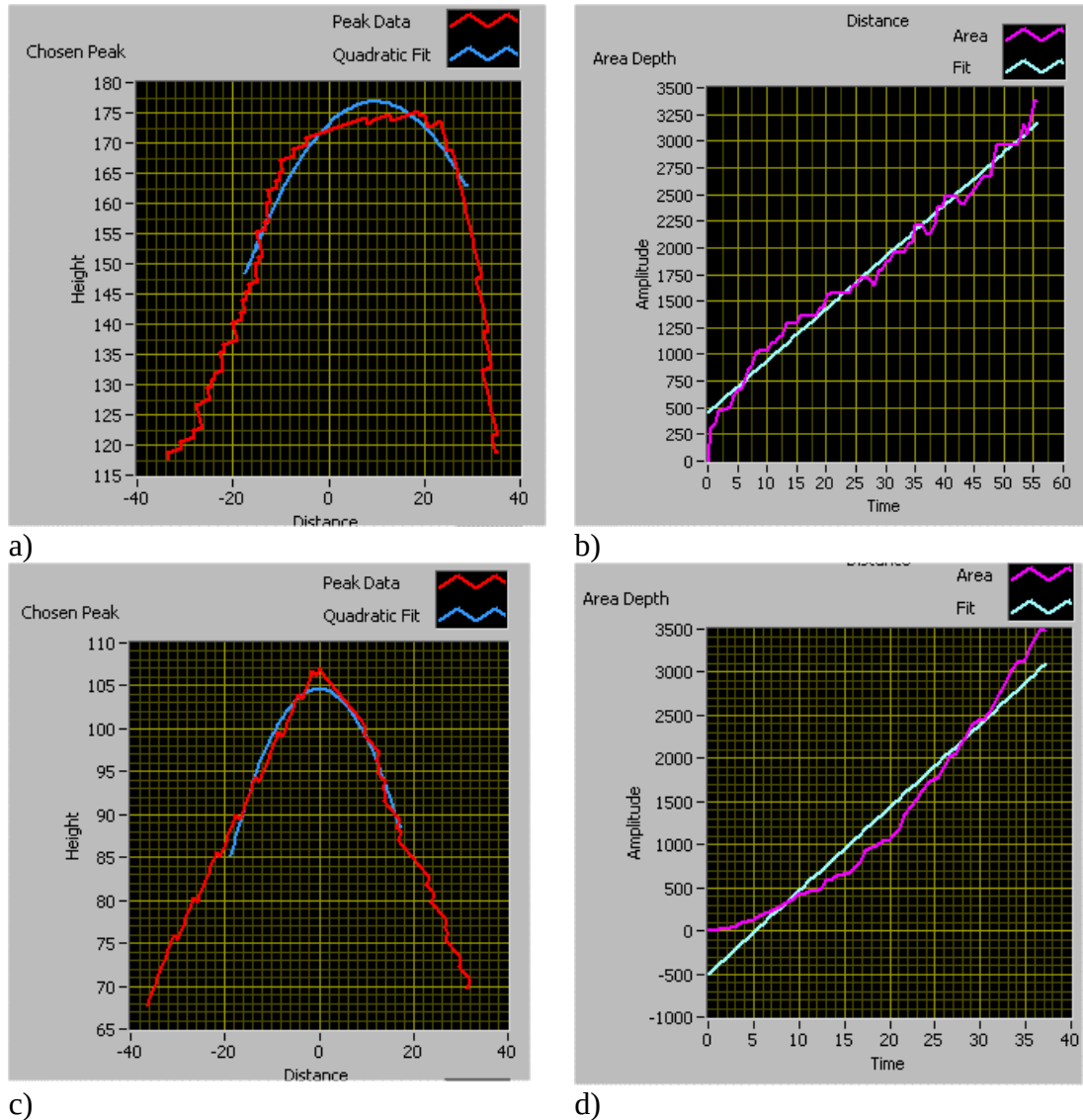


Figure 14, Typical peaks showing relatively poor fits, a) peak flatter than fitted parabola, b) area-depth curve and linear fit corresponding to a), c) peak sharper than fit, d) linear fit corresponding to c)

The FFT, Figure 13c, is also characteristic of the deviation from a round particle, as the FFT of a horizontal line unwrapped profile derived from a round particle would be a delta function showing a DC signal. Thus the sharper the slope of the FFT, the closer the particle is to a round particle.

The parabolic fit is an attempt to describe the shape of specific asperities. In many cases the fit is a reasonable description of the asperity, but in others the parabola is sharper or flatter than the real asperity (Figure 14).

The overall results are shown in Table 3. This shows the overall size of the particles that is comparable to that determined earlier by image analysis. Some differences in the average parameters between the F30 alumina and the 25-36 sand can be seen, but the differences are within the standard deviation.

Table 3, Parameters determined by unwrapping.

	Radius, μm	Parabolic Fit	F ratio	A-d Slope
F30 Alumina	630 $\pm$ 138	-0.021 $\pm$ 0.01	14.3 $\pm$ 4.3	251 $\pm$ 151
25-36 Sand	688 $\pm$ 95	-0.013 $\pm$ 0.01	16.15 $\pm$ 2.9	357 $\pm$ 182

4 DISCUSSION AND CONCLUSIONS

A wide range of techniques have been used to analyse the size and shape of a range of abrasives used in abrasion testing of a range of hardmetals. The determination of particle size showed reasonable comparability between sieving analysis and the image analysis of particles. As expected, the sizes determined from a cross-sectional analysis were significantly reduced from those from images obtained from silhouettes.

A number of different methods for determining shape were used including the SPQ parameter that has been one of the more successful shape parameters to be described so far. Other parameters were defined in an unwrapping analysis. Only small differences in the shape parameters were found between sand and alumina abrasives that were within the standard deviation of the methods.

The use of confocal scanning optical microscopy was shown to be ideally suited to analyses the 3D shape of single particles, but reinforced the point that every particle had a distinctly different shape.

This was a major difficulty that applied to most of the measurements; the spread in test parameters from one asperity to another and from one particle to another was always much larger than any change from one type of abrasive to another.

The exception to this was in the analysis of sand abrasive that had been used in the ASTM B611 test. Here the sand that had passed through the test system had been broken down to a particle size that was more than ten times smaller than the original size. There was also a change in the particle shape that had become much sharper. This was distinguished well by the shape parameter measurements that were made.

5 ACKNOWLEDGEMENTS

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6 REFERENCES

- 1) Hutchings, I. M., Tribology: Friction and wear of engineering materials, Edward Arnold, 1992
- 2) Wadell, H, Volume, Shape and Roundness of Rock Particles, J Geol, 40(1932)443-451
- 3) Radnui, S and Roylance, B, The classification of wear particle shape, Lub Eng, 51(1955)432-437

- 4) Meloy, T P, Particulate Shape Characterisation, J Powd Bulk Solids Tech 2(1978)13-23
- 5) Swanson, P A and Vetter, A F, The Measurement of Abrasive Particle Shape and its Effect on Wear, ASLE Trans, 28(1984)225-230
- 6) Lees, G, The Measurement of Particle Shape and its Influence in Engineering Materials, J British Granite and Whinestone Federation, 4(1964)1-22
- 7) Palasamudram, S L and Bahadur, S, Particle Characterisation for Angularity and the Effects of Particle Size and Angularity on Erosion in a Fluidized Bed, Wear, 203-204(1997)455-463
- 8) Hamblin, M G and Stachowiak, G W, A Multi-Scale Measure of Particle Abrasivity and its Relation to Two-Body Abrasive Wear, Proc of the 4th International Tribology Conference, Perth, Australia, Austrib 94, Frontiers in Tribology, 1(1994)191-198
- 9) Hamblin, M G and Stachowiak, G W, Description of Abrasive Particle Shape and its Relation to Two-Body Abrasive Wear, Tribology Transactions, 39(1996)803-810
- 10) Stachowiak, G B, and Stachowiak, G W, The Effects of Particle Characteristics on Three-Body Abrasive Wear, Wear, 249(2001)201-207
- 11) Vespui, M A, van der Velden, P de With, G, and P J Slikkerveer, P J, Wear 199(1996)122-126
- 12) ASTM B611-85, Standard test method for the abrasive wear resistance of cemented carbides, Annual Book of ASTM Standards, 02.05(1995)326-327
- 13) N J McCormick and M G Gee, The Measurement of the Shape of Abrasive Particles with Confocal Optical Microscopy, NPL Report
- 14) ImageJ Image Analysis Software, <http://rsb.info.nih.gov/ij/>