

NPL REPORT
DEPC MPE 010

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Shape of Abrasive Particles
with Confocal Optical
Microscopy**

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The Measurement of the Shape of Abrasive Particles with Confocal Optical Microscopy

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Engineering and Process Control Division

ABSTRACT

This short communication describes the application of confocal optical microscopy to the evaluation of the shape of abrasive particles.

The confocal microscope was found to be ideally suited to the analysis as it naturally produces height data with near optimum resolution for shape analysis.

Preliminary results are given on the analysis of two abrasives.

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ISSN 1744-0262

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Hampton Road, Teddington, Middlesex, TW11 0LW

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We gratefully acknowledge the financial support of the UK Department of
Trade and Industry (National Measurement System Policy Unit)

Approved on behalf of the Managing Director, NPL,
by Dr G Sims, Knowledge Leader, Engineering and Process Control Division
authorised by Director, Engineering and Process Control Division

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1 BACKGROUND

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 by simple shape analysis [1-3], Fourier analysis [4-5], analysis of the sharpness of asperities on the particles, and the curvature of the particle outline [6-10]. 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 shape information, other techniques are required. The height resolution, lateral resolution and range of confocal optical microscopy are ideally suited to the analysis of abrasive particle shape. The resolution and range are dictated 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 .

2 EXPERIMENTAL METHOD

2.1 MATERIALS EXAMINED

Two abrasive grits were examined. These were alumina and silica abrasives used in the ASTM B611 [12] and other abrasive tests [Figure 1]. The size and overall shape of the particles was measured by combined microscopy and image analysis. The factors that were measured were the circularity (defined as $4A\pi/P^2$) where A is the projected particle area and P is the projected particle perimeter), the equivalent circle diameter and the ratio of the maximum and minimum diameters of the particles. Both the ratio and the circularity are factors equal to 1 for a circle and decrease as the angularity of the particle decreases. About 70 particles were measured for each abrasive.

Table 1 gives the results of these measurements. It can be seen that the alumina abrasive had an average equivalent size of 649 μm compared with the smaller size of 490 μm for the silica abrasive. Both the ratio and the circularity parameters were significantly larger for the silica than the alumina, in line with the appearance of the particles. However, the difference in the ratio of maximum to minimum diameter parameter is lower than that for the circularity factor.

Table 1 Simple size and shape analysis of particles

	Equivalent Circle Diameter, μm	Ratio of Maximum to Minimum Diameter	Circularity
Silica	489.9 $\pm$ 36.8	0.74 $\pm$ 0.11	0.77 $\pm$ 0.04
Alumina	649.4 $\pm$ 89.1	0.71 $\pm$ 0.13	0.70 $\pm$ 0.06

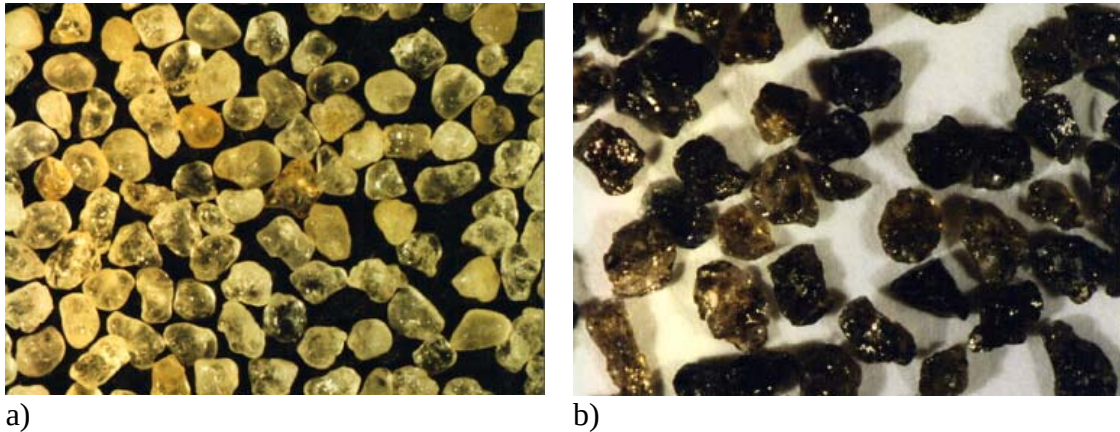


Figure 1 Optical micrographs of abrasives, a) silica, b) alumina. Note that these images are optimised for conventional microscopy and use contrasting backgrounds.

2.2 CONFOCAL MICROSCOPY

For both abrasive grit samples small amounts of powder were sprinkled on to a flat substrate at a density low enough that the majority of particles were separated. The specimen then had a sputtered gold coating applied so that any particle translucency was minimised. As the abrasive particles in a sample all have slightly different sizes and shapes, it is important to make measurements on a reasonably large number of particles for each sample. The principle that was used in this study was to image the particle as they naturally lay on the substrate, analysing the asperity whose tip formed the closest point of approach to the microscope lens.

Each separated particle was then imaged using a Tracor Northern Tandem Scanning microscope. This microscope is a confocal scanning optical microscope (SOM) that uses pinholes accurately positioned in a spinning disc to provide illumination and imaging apertures. As the disk spins the pinholes move in spirals and form a full field image (Figure 2 below shows an idealised confocal microscope). By using pinholes for illumination and imaging, the confocal microscope captures only light from close to the plane of focus. In the tandem scanning microscope a beam splitter allows the same lens to be used for both illumination and imaging. The depth of the image slice produced depends on the numerical aperture of the lens and in this study an 8x magnification objective was used with a numerical aperture of 0.2, which results in a image slice of approximately 4 μm in depth.

Acquiring these image slices, whilst changing the height of the objective using a piezoelectric actuator, allows a stack of image slices to be created. It is then straightforward to process the image stack and produce a simple image, that is everywhere in focus, as well as a height map corresponding to the vertical focal position in the image stack, which has the maximum intensity for a given pixel. The image slices were acquired with a separation of 2 micrometres.

There was a limit of 400 μm in vertical movement for the objective, which meant that in some peripheral parts of the image the surface of the particle or the substrate was

never encountered. This resulted in image noise at these positions as there was not enough information to determine a surface level. Additionally any surface profilometry technique, such as SOM, has limitations on the maximum angle of surface that can be reliably measured and this leads to loss of signal and consequent noise in the final image.

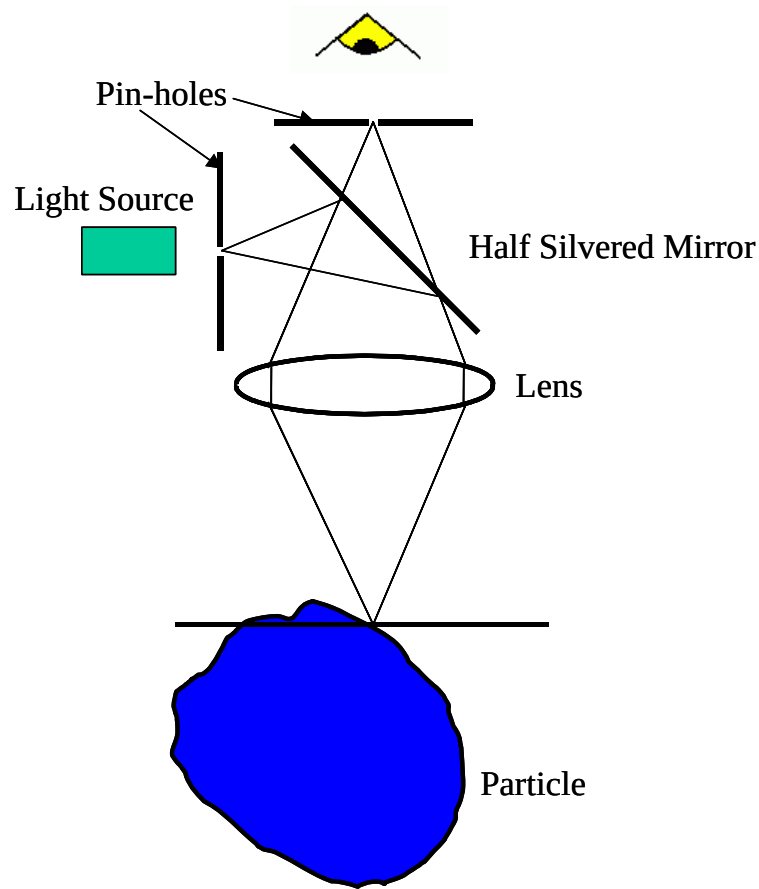


Figure 2 Schematic diagram of the imaging process for a confocal microscope

The objective lens was chosen so that an image of a single particle filled a single field of view. In any image slice the region of interest in the image was defined by using a semi-automatic image processing script that was run using the ImageJ package [13]. This script identified the boundary of the particle either using thresholding on the in-focus image, thresholding on the height map or thresholding based on variance filter processing of the height map. There was user choice of which of these three algorithms best identified the particle boundary.

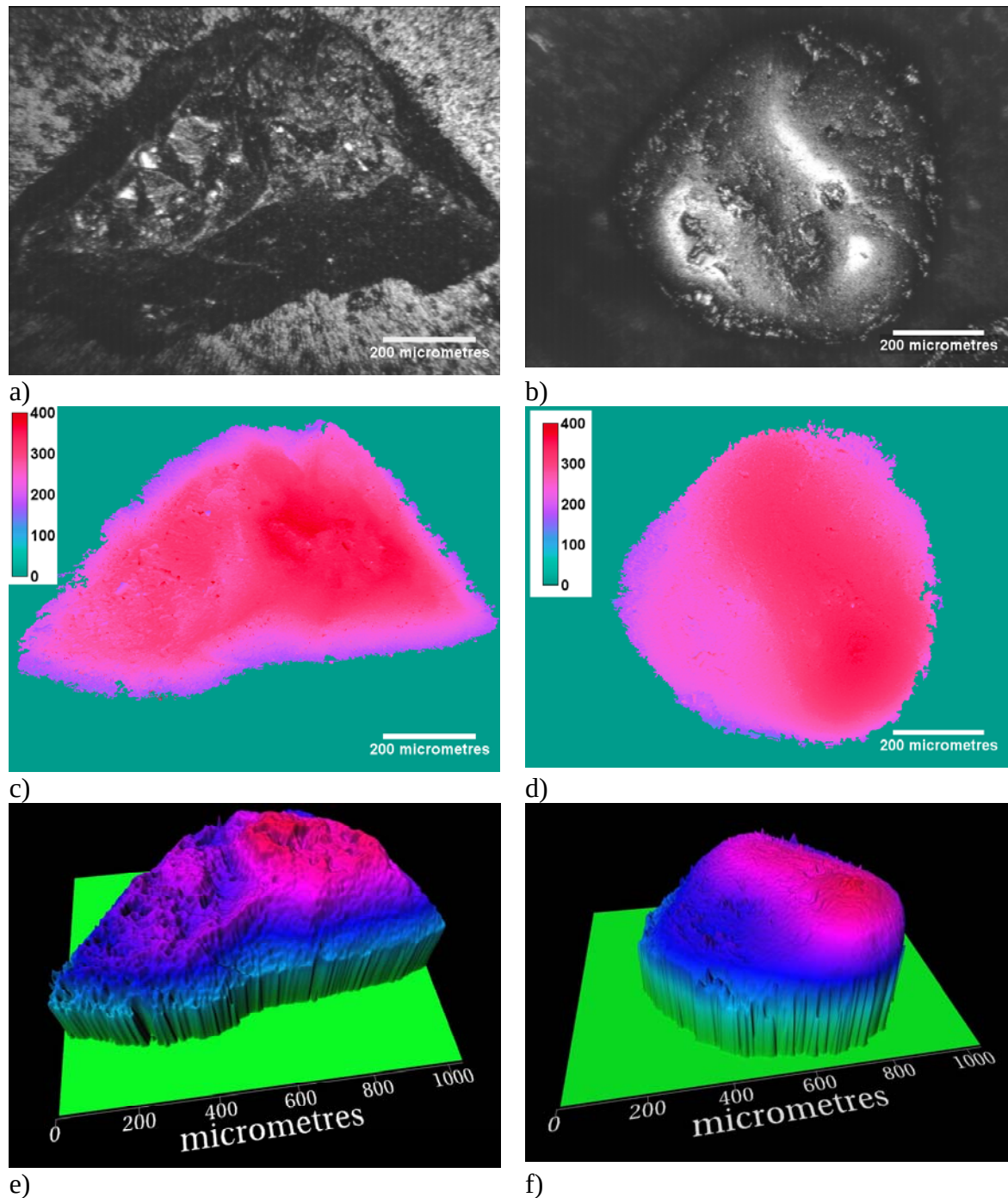


Figure 3 Results of confocal optical microscope measurements on single particles, a) image of alumina particle, b) image of silica particle, c) height map for particle in a) (height scale in micrometres), d) height map for particle in b) (height scale in micrometres), e) surface reconstruction for particle in a), f) surface reconstruction for particle in b).

Subsequent processing was then carried out if the boundary of the particles intersected the edge of the image. In this case the thresholding level was sequentially lowered in steps and thresholding applied to the height map image. This was done until the perimeter of the thresholded area just touched the edge of the image. This threshold level was then incremented one step and this new region of interest was saved as an

image and used in the subsequent analysis, all images shown in this publication were processed in this manner.

Typical particle images and their corresponding height maps are shown in Figure 3. Figures 3a and 3b 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 3e and 3f for the two different powders.

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 visually to be more angular than the 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 5a and 5b.

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 could be made for each curve.

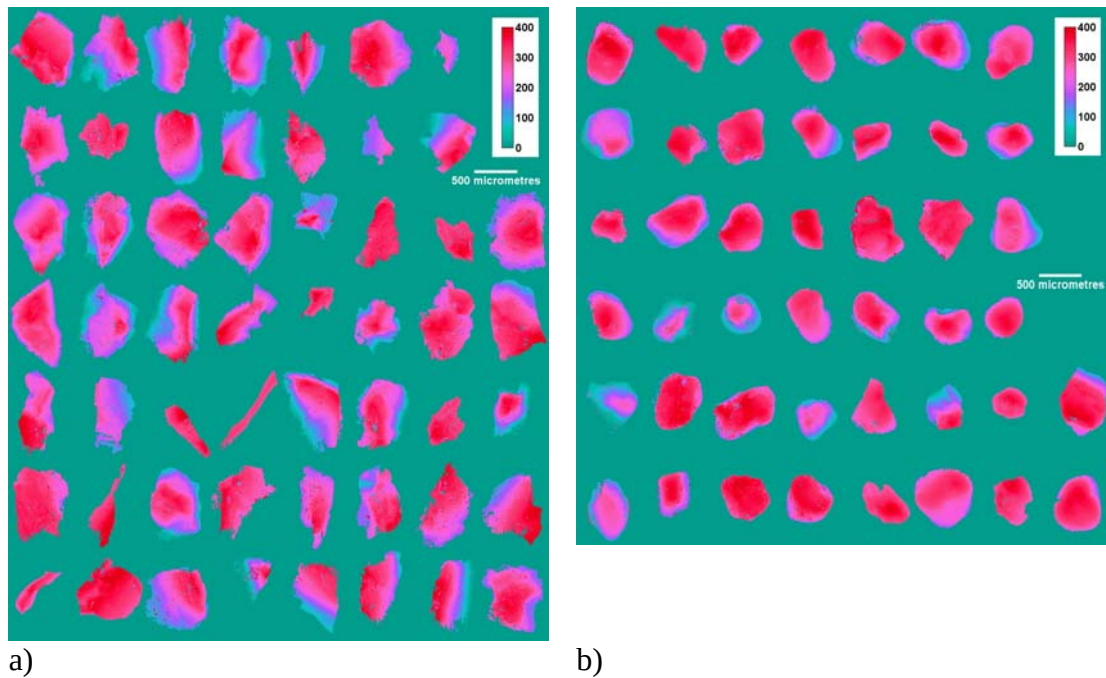


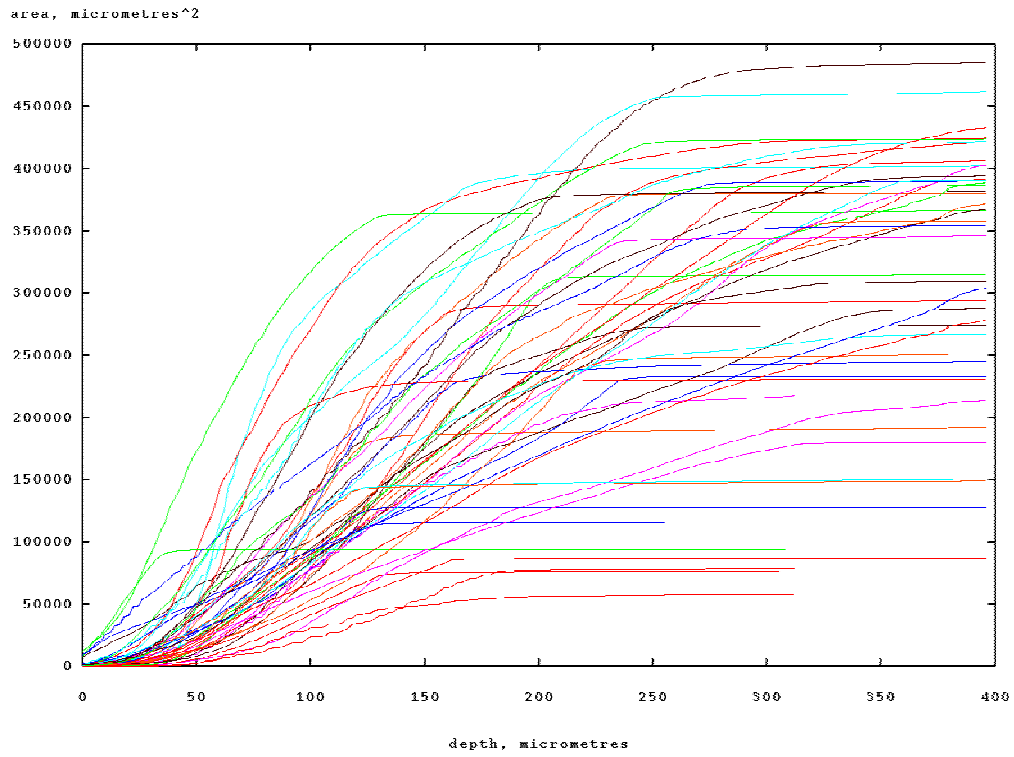
Figure 4 Montages of all particles analysed in this study, a) alumina particles, b) silica particles.

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 5c and 5d.

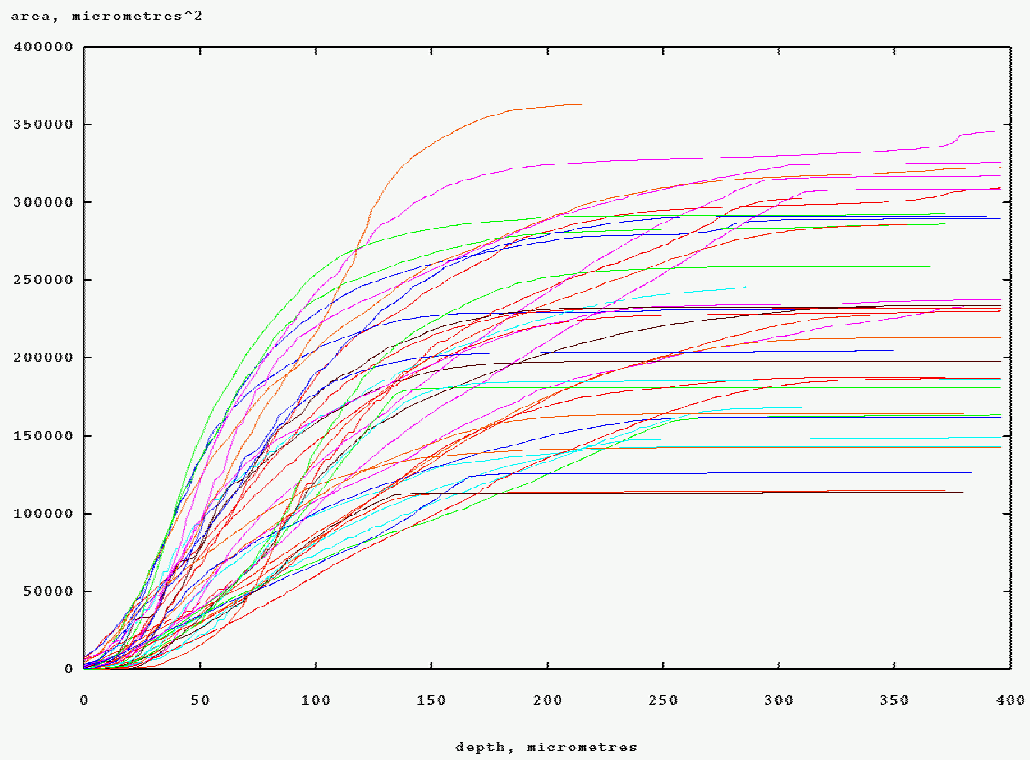
3 DISCUSSION

3.1 INSTRUMENTAL CONSIDERATIONS

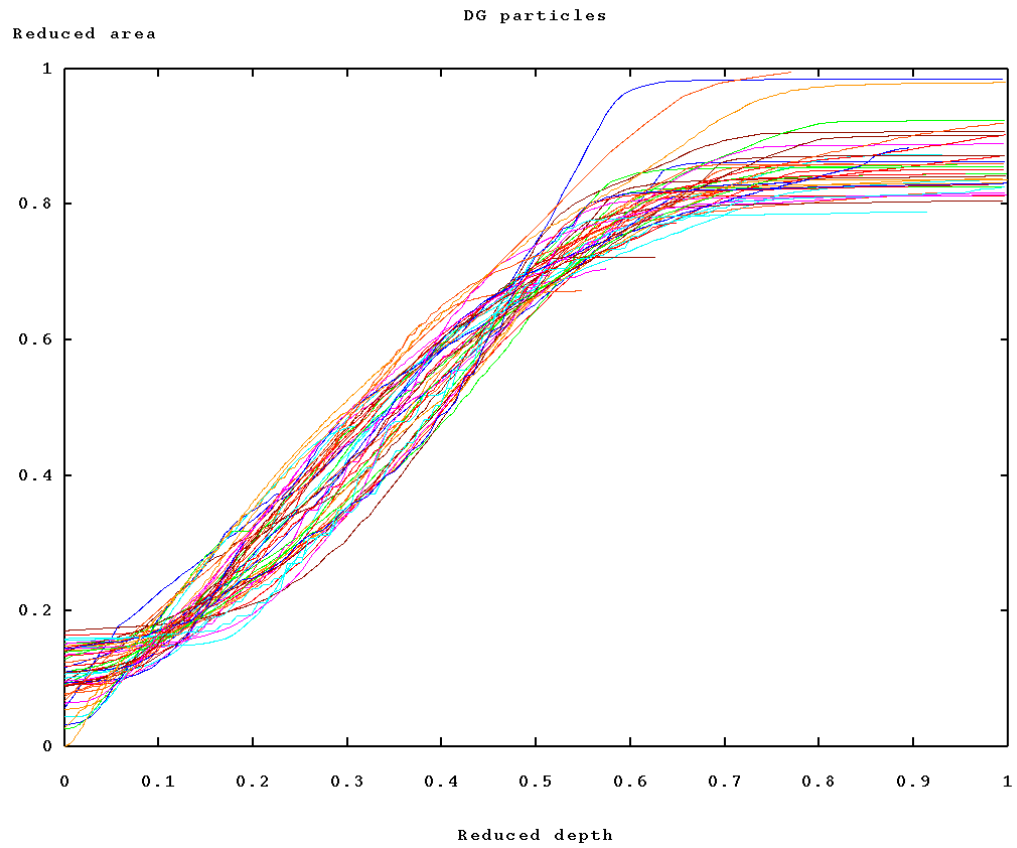
Height noise is found in the images due to various sources. As mentioned before there may be slopes that are too steep to successfully image, because insufficient light is reflected back towards the lens. Also there can be undesirable lensing effects caused by specimen translucency. The lensing artefact may be minimised by coating the specimen, although the gold sputtered coating may cause less light to be returned from steeply sloping surfaces and a more diffuse coating may be more successful. The height noise was identified using image analysis techniques, although this may be more easily accomplished in the future by developing the SOM acquisition software to identify this effect when capturing images. To repair the gaps in the images a spline surface-fitting algorithm was utilised. This allowed small patches to be removed from the image and using a mask, the missing surface was interpolated. When all the patches had been analysed the whole image was reconstructed from the new surface interpolations patches. This proved successful for trial images but required considerable computation and was only used for the images in Figure 3.



a)



c)



c)

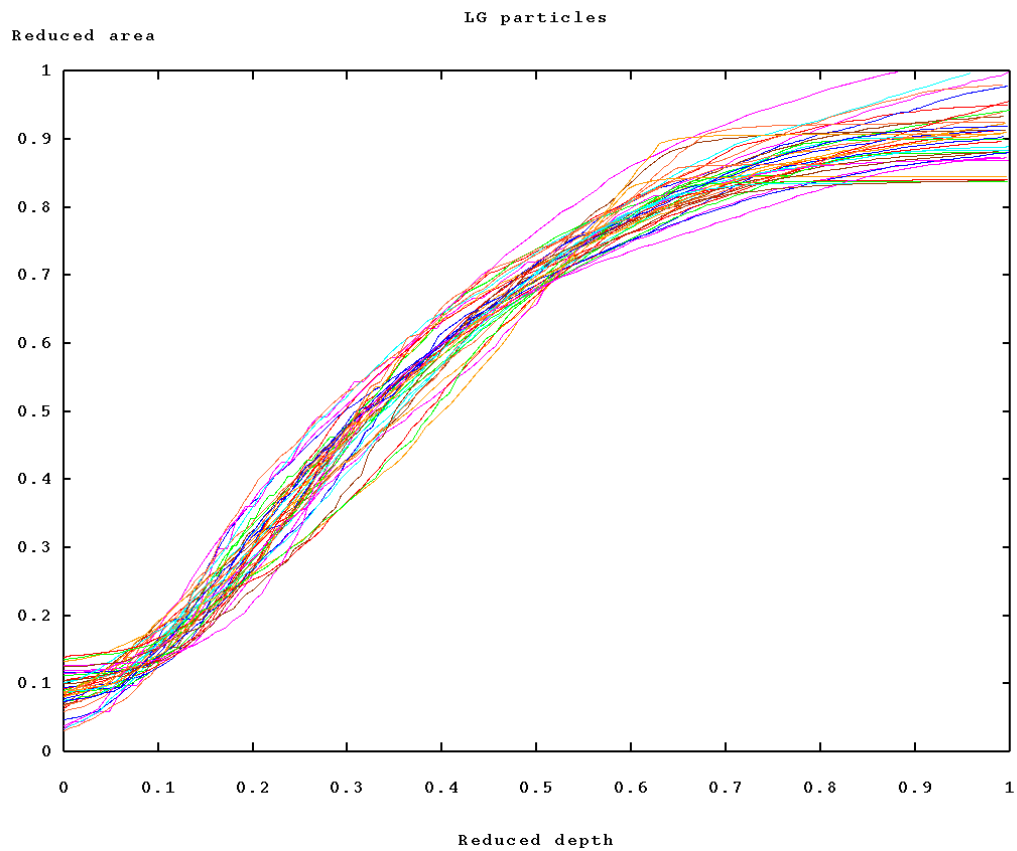


Figure 5, Area-depth curves for particles, a) alumina, b) silica, c) normalised (reduced) alumina, d) normalised (reduced) silica

3.2 PARTICLE SHAPE MEASUREMENT

The results of the confocal scanning optical microscope examination show that it is ideally suited to examining the shape of individual abrasive particles. The images and height maps of individual particles show clear differences between the alumina and silica particles in terms of the roundness of the particles and the angularity. A major advantage of the technique is that data is obtained from particles naturally orientated giving information on the area of particles at any depth. The area-depth curves formed summarise the geometrical shapes of the asperities of the particles. However, in practice it was found that there was considerable variation in these curves for particles within either of the two samples, making it difficult to assess the differences between the two abrasives.

Two approaches were used to extract some useful data from these analyses. The first was to fit an ellipsoid of revolution to the asperity area depth data. This gave tighter families of curves for each of the two abrasives. It was difficult to see much overall difference in shape between these families of curves. However, the fits to the data enabled a sharpness parameter S for each particle to be defined as

$$S = \frac{\sqrt{ac}}{b}$$

This is in essence the ratio of the width of the fitted ellipsoid to the height from its midplane. The average value of this sharpness parameter was 0.98 ± 0.51 for the alumina abrasive, and was 1.19 ± 0.41 for the silica abrasive. For a sphere ($a=b=c$) the value is 1. Thus the silica particles are, on average, flatter than a spherical cap, and the alumina particles are on average spherical.

The second approach was to consider how these particles would cause damage in an abrasion event. Two parameters can be easily derived from the 3D data are the area of the asperity at a given depth and the physical slope of the asperity at that depth. The area of the asperity at a particular depth dictates how deeply the asperity will sink into the surface dictated by the local hardness. This parameter is likely to be most important for materials that undergo fracture in abrasion. The slope is a parameter that will control wear in materials undergoing plastic deformation in abrasion. The measures were evaluated at constant depth and showed that there was a considerable difference between the parameters for the two particle types (Tables 2 and 3), with the alumina abrasive always sharper and more penetrating than the silica abrasive (Figure 6).

Table 2 Showing variation in average area of alumina and silica particles examined at a 20 and 40 μm depth

	Mean Area, μm^2	Standard deviation, μm^2
Alumina particle at 20 μm depth	9146	12665
Alumina particle at 40 μm depth	25097	25102
Silica particle at 20 μm depth	16496	11857
Silica particle at 40 μm depth	50501	28815

Table 3 Showing variation with average slope of equivalent radius versus depth for alumina and silica particles at a 20 and 40 μm depth.

	Mean slope	Standard deviation
Alumina particle at 20 μm depth	1.46	0.82
Alumina particle at 40 μm depth	1.93	0.92
Silica particle at 20 μm depth	3.27	1.26
Silica particle at 40 μm depth	1.91	0.81

3.3 CORRELATION WITH WEAR RESULTS

The results of Figure 7 show that wear from the alumina abrasive is about 10x as severe as the wear from the silica abrasive. One of the reasons for this difference is likely to be the difference in shape of the two abrasives that was observed by the two area-depth parameters, but this difference in wear is also due to many other factors. Thus the hardness of the alumina abrasive is about twice that of the silica (2300 HV0.2 versus 1100 HV0.2). However, this hardness difference does not explain the large difference in wear that is seen. Another effect that does occur is the breakdown in the particles in the abrasion process where it is known that the silica breaks down much more than the alumina. However, the effect of this on the wear that is produced is not known.

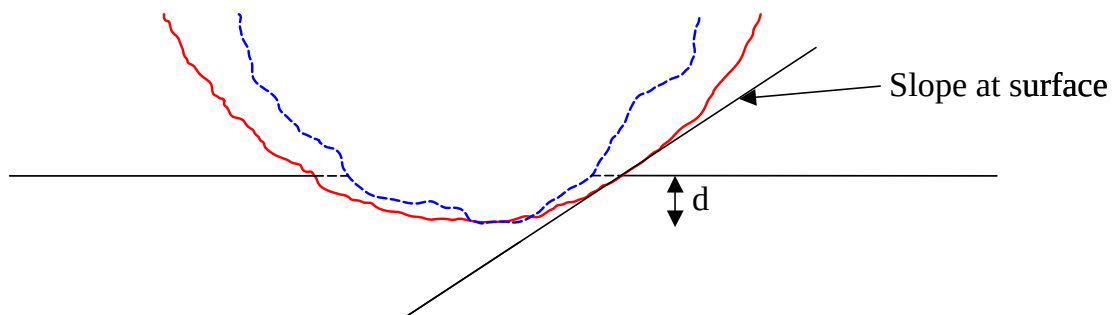


Figure 6 Schematic showing penetration of asperities on particles into surface. Blue is alumina, red is silica.

3.4 SUGGESTIONS FOR FURTHER WORK

The results reported in this preliminary study show a clear difference in the shape of the two different abrasives. However, the effect on wear is not clear due to the fact that the abrasives were from different materials. Further work needs to be carried out both with respect to wear testing and also with respect to SOM shape analysis of abrasive form where the two different abrasives are from the same material and thus the same hardness, but have radically different shapes.

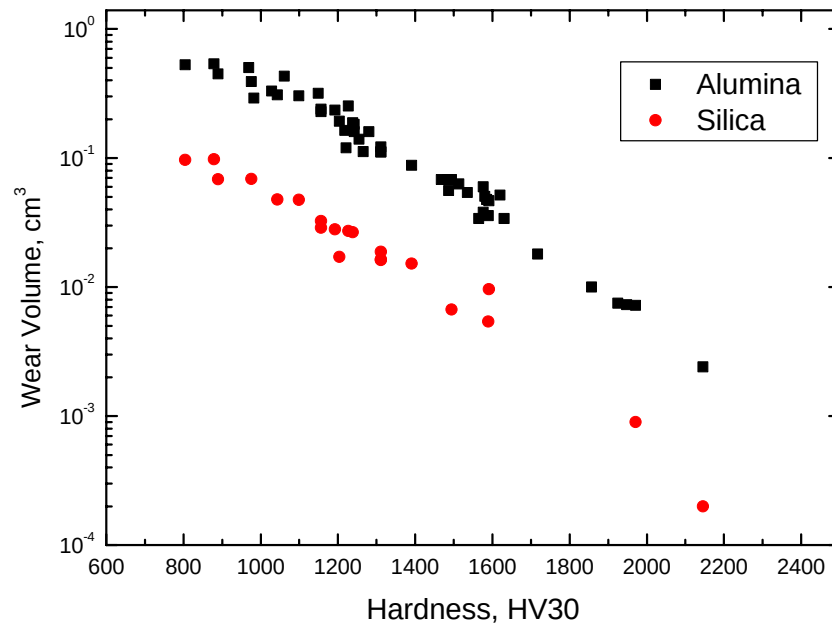


Figure 7 ASTM B611 abrasive test results on range of WC/Co hardmetals for silica and alumina abrasives examined in this study.

It should be noted that the SOM data is a full 3D data set for asperities on the different particles. The capture of the three dimensional profile potentially allows some interesting comparisons to be made with analysis of the sharpness of particles such as the SPQ analysis [8-10]. For a given particle it would be possible to synthesise many SPQ sections around a peak and explore the inherent variation in this or other sharpness parameters.

4 CONCLUSION

It has been shown that the confocal scanning optical microscope can be used to provide a very effective method for the analysis of the shape of abrasive particles. Much larger differences in shape between the two abrasives were found for parameters based on area depth curves than for simple image analysis based shape factors.

5 ACKNOWLEDGEMENTS

The authors would like to acknowledge the support of DTI for this work through its Materials Measurement Programmes.

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) ImageJ Image Analysis Software, <http://rsb.info.nih.gov/ij/>