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The Measurement of Uncertainty in Grain Size Distribution

Interlaboratory Exercise Part 1 - Reference Images

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ABSTRACT

An investigation of the grain size distribution by the intercept method, in three engineering metallic materials, an Al alloy (AA5182) and two Ni alloys (Waspaloy and IN909) has been carried out through an interlaboratory exercise, ILE. The ILE was performed in two parts. In the first part reference images generated at NPL from optical micrographs were measured. In the second part, a set of optical micrographs were measured. This report concerns the first part of the ILE, the measurements on the reference images.

The ILE was supported by measurements of intercept distributions obtained from a 3-D model shape, a tetrakaidecahedron. A PC computer program was written to rotate and randomly section the model generating data on section area, intercept lengths, and area sides and vertices. Comparisons were made between the model distribution data and the distribution data from the material microstructures.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
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1 INTRODUCTION

With DTI support NPL is working in collaboration with a number of partners from industry and academia (see Appendices A and B for contact lists) to examine the uncertainties associated with measuring grain size distributions in representative engineering alloys through the use of structured interlaboratory exercises (ILEs).

The method used was based on the line intercept technique.

The exercise was performed in two parts:

- Part I - Measurement of Reference Images
- Part II - Measurement of Material Microstructures

This report, CMMT(A)154, is concerned with Part I. A further report CMMT(A)N2 provides information on Part II.

For the ILE in part I thirteen sets of reference images and instructions were circulated to an inner circle of direct participants (APPENDIX A) who returned measurement data to NPL for analysis. Additional sets were circulated for information to a further twenty correspondents (APPENDIX B) including the DTI project monitor, Prof A W D Hills.

A discussion of grain size distribution measurement methods is given in section 4.

The measurement exercise was supported by modelling studies using 2-D sections taken from a 3-D distribution of grains. A tetrakaidecahedron was used as the model shape (section 5).

The results of the exercise are being used to support the development of a Good Practice Guide for the measurement of a mean value of grain size in a parallel project, MMP7, being undertaken by AEA Technology and Sheffield Hallam University (see APPENDIX C for contact list).

There is a generic requirement for improved guidelines for the measurement of grain size distribution in a wide range of industrial materials. The properties of metallic products are frequently controlled by the grain size and size distribution of the underlying microstructures. UK industry has to meet severe international competition and steady improvements in consistency and performance are needed each year to maintain and enhance competitiveness. Standardised methods of measurement of structure are required to ensure that materials with repeatable properties are produced and a link between processing and structure reinforced.

It is widely recognised by industry that uniformity or otherwise of grain size distribution is an important feature of the microstructure that controls properties. In both single and multiphase materials the measurement of an average value of the grain size is vital and there are existing and widely used standards for this purpose, such as ASTM E112, E1181 and E1382. However, although there are routines in ASTM E1181 for the measurement of size distribution these are not widely agreed within UK industry and there is a need to improve measurement practice.

1. TERMINOLOGY AND STANDARDS

The relevant microstructural standards are:

ASTM E112	Grain size Heyn - linear Jeffries - circle
ASTM E1181	Duplex Grain Sizes
ASTM E1382	Grain Size using Image Analysis
ASTM E930	Largest Grain Size
prEN 861	Ceramic grain sizes

Standard methods are adequate for the measurement of a single average value of grain size in materials where there are reasonable preparation methods for discriminating different grains. However, there are still many important engineering materials which are extremely difficult to prepare clear images for measurement. Also the current standards are quite detailed and there is scope for a more straightforward technical guide.

A primary technical issue is a requirement for a quantitative evaluation of the uncertainties associated with measuring grain size distributions. This will enable material specifications to be developed that will use unambiguous definitions to the benefit of both customer and supplier. This is a generic requirement relevant to a broad range of material types.

ASTM E1181 addresses some of the issues, for example it discusses various typical examples, such as:

- isolated coarse grains in finer grained matrix
- extremely wide distributions
- bimodal distributions

and mentions other difficult issues, such as:

- banded structures, necklace structures and systematic variations across sections of products.

ASTM E1181 also contains comparison charts for estimating area fractions (possibly in multiphase materials). A number of procedures are defined, including:

- a) test grid on transparent overlay - superimposed image - estimate area fractions
- b) largest grains
- c) comparison charts
- d) intercept method for grain size.

The standard advises that the estimation of area fractions of different sizes is subjective and prone to error.

The standards recommend that the most effective method for measuring intercept distributions is to use a semi-automatic image analysis system with digitising tablets and electronic pencils/cursors - using a test grid with 5 evenly spaced horizontal lines. The intercepts are classified by size and the data is presented as a histogram or frequency plot.

An industrial partner has itemised a number of confusing requirements arising from the use of ASTM E1181:

- a) Grain size ASTM 5 or coarser.
- b) Grain size ASTM Index No 5 or greater.
- c) Grain size ASTM 5, occasionally ASTM 0.
- d) Grain size ASTM 4, some ASTM 3

2 MATERIALS

Reference images were prepared (Figs 1-3) from three materials provided by ILE participants. The method for preparation of the images is reported separately [1].

Al alloy	AA5182	Alcan International Nominally equiaxed structure
Ni alloy	Waspaloy	Incotest Wide grained alloy
Ni alloy	IN909	Incotest Wide grained alloy

3 ILE PARTICIPATION AND PLAN

The following organisations contributed to part I on reference images:

Organisation	Contact	Abbreviated name
Alcan International	J Brown	ALCAN
Doncasters	J Brooks	DONCASTERS
Firth Rixon Superalloys	L Rowland	FIRTH RIXON
Incotest	I Elliott	INCOTEST
London & Scandinavian Metallurgical Co Ltd*	E Birch	LSM
London & Scandinavian Metallurgical Co Ltd*	E Birch	LSM/PB
London & Scandinavian Metallurgical Co Ltd*	E Birch	LSM/SM
National Physical Laboratory	E Bennett	NPL
Rolls Royce	G Baxter	ROLLS ROYCE
Sheffield Testing Laboratories	G Black	STL
Sheffield Hallam University	J Cawley	Sheffield Hallam
Alstom	G McColvin	ALSTOM

*Three sets of measurements were provided by 3 different operators at LSM.

The exercise was carried out in two parts

Participants made measurements on a reference line on each reference image (Figs 1-3)

Participants made measurements along lines on each image that they drew themselves.

Magnifications were calculated using images of a graticule of a given length taken under the same conditions as the reference images.

A full instruction pack sent to each participant is included in APPENDIX D.

4 GRAIN SIZE DISTRIBUTION METHODS

4.1 PROPERTY/DISTRIBUTION DEPENDENCIES

The type of distribution measurement to be preferred depends on what effect a specific distribution of sizes has on properties. At present it is not known whether the properties are determined by the number fraction of grains of a given size or by length fraction, by area fraction, or by volume fraction, although there is some evidence that volume fraction (size) determines hardness in Powder Metallurgy products [2,3].

Material structures are generally characterised, apart from their chemical composition, by a size parameter, S . In single phase materials the most commonly used size parameter is grain size, d , which has the dimensions of length. Because structures are usually measured on a two-dimensional polished surface it is common practice to measure grain size by the length of intercept, d , within each grain of a line superimposed across the image of the microstructure.

Many intercepts are measured on random lines and the number average is taken to give an arithmetic mean linear intercept, d_m .

$$d_m = \frac{\sum d_i}{N} \quad (1)$$

where N is the total number of intercepts and d_i are individual intercept lengths. This value in many cases is taken to be the grain size without converting it to a true 3-D grain diameter, that requires the use of a multiplication factor dependent on the shape of the individual grains.

Property models are generally related to the size parameter, S , by equations of the type

$$P = a + bS^n \quad (2)$$

where a , b and n are constants and P is the property of interest. For example in many metallic materials the Hall-Petch equation can be used to predict the effect of grain size, d , on strength or hardness, H , with

$$H = H_0 + E_1 d^{-1/2} \quad (3)$$

Thus, expression (3) shows by comparison with expression (2) that $a = H_0$, $b = E_1$ and $n = -1/2$.

When models of these types are compared with microstructural measurements the grain size is frequently taken to be the arithmetic (number average) mean linear intercept, d_m , obtained from a two-dimensional image. Because all single-phased materials have microstructures in which the grains are distributed in size, a more general form for expression (3) can be derived. Also the structures of single-phased materials can be characterised in several ways other than by using a number distribution of d_i . For example, the grain area or volume distributions can be obtained on a size basis, by line, by area fraction or by volume fraction. All these different methods of structure characterisation can have different consequences for the predictive models described above since they lead to different integrals which have to be evaluated.

The most common technique is to use a number distribution of size $f_n(S)$; this is frequently found to be lognormal for intercepts. However, a different distribution can be developed based on the size fraction $f_s(S)$ rather than number. Size fraction is obtained by dividing the size of interest by the total size and the size fraction distribution is derived by plotting the size fraction against size.

Thus, there are six possible distributions, three of number and three of size, based on intercept, area or volume. These distribution functions are

Number - $f_n(I)$, $f_n(A)$ and $f_n(V)$

Size - $f_s(I)$, $f_s(A)$ and $f_s(V)$

Some of these distributions (ie $f_n(I)$ and $f_n(A)$) can be measured directly but some types of distribution, ie $f_n(V)$ and $f_s(V)$, must be calculated from directly measured distributions, usually by making assumptions about grain shape. The number distributions are often approximated by a lognormal function. A lognormal distribution $f[x]$ is defined as:

$$f[x] = \frac{1}{x \sigma \sqrt{2\pi}} \exp \left[-\frac{(\ln(x) - \ln(\mu_g))^2}{2\sigma^2} \right] \quad (4)$$

where x is the variable, σ is a measure of the distribution width and μ_g is a measure of the distribution mean.

The lognormal distribution parameters, μ_g and σ (the geometric mean and standard deviation) can be ascertained by plotting the measured linear intercept data on lognormal probability axes, with a \log_{10} abscissa. The arithmetic number mean is higher than the geometric mean and the amount by which it is higher is dependent on the distribution width, σ .

At present the arithmetic number mean is probably the best value to recommend as an indicator of mean size, as this is the value most widely measured and quoted in research papers. It is not recommended at this stage that this 2-D mean intercept value is converted to a value equivalent to that from a 3-D structure.

4.2 DISTRIBUTION TYPES

Intercept data can be plotted systematically by a number of methods in order to compare and contrast the different ways in which distributions of sizes can be displayed and examined [2-7], in particular by using distributions based on either number or size probabilities.

For example:

the data can be shown as cumulative probability plots with the ordinate (y) given a linear scale or by using probability paper. The latter method expands the extremes of the distribution and contracts the central regimes.

- the data can be plotted as cumulative probability plots with the abscissa (x) on a linear or logarithmic scale.

a further refinement is to normalise the abscissa values, ie normalised intercept or normalised area. This is obtained by dividing by the number or size average.

the data can be plotted as intercepts, or areas, either by number (A) of the i^{th} ranked data plot or as cumulative length of intercept (B) or cumulative area (C). Area can be estimated by squaring the intercepts. For A , B and C the mathematical expressions used to calculate cumulative probability, P_i are given by

Number Distribution:

$$P_i = \frac{\sum_{i=1}^i n_i - 0.5}{N_d} \quad (\text{A) for intercepts} \quad (5)$$

$$P_i = \frac{\sum_{i=1}^i n_i - 0.5}{N_A} \quad (\text{A) for areas or estimated areas} \quad (6)$$

Size Distribution:

$$P_i = \frac{\sum_{i=1}^i d_i}{\sum_{i=1}^N d_i}, \quad (\text{B) for intercepts} \quad (7)$$

$$P_i = \frac{\sum_{i=1}^i A_i}{\sum_{i=1}^N A_i}, \quad (\text{C) for areas or estimated areas} \quad (8)$$

where N_d is the total number of intercepts, d_i , counted

N_A is the total number of grain areas, A_i , counted

If area is estimated from intercepts squared then $N_d = N_A$

$\sum_{i=1}^i n_i$ is the cumulative number of intercepts or areas up to the i^{th} data point

$\sum_{i=1}^i d_i$ is the cumulative length of intercept up to the i^{th} data point

$\sum_{i=1}^N d_i$ is the total cumulative length of intercept

$\sum_{i=1}^i A_i$ is the cumulative area up to the i^{th} data point

$\sum_{i=1}^N A_i$ is the total cumulative area.

There are several questions to be asked of these different types of data plots.

- *Do the data reveal differences more clearly when plotted using a linear abscissa rather than logarithmically? Are there advantages in using normalised data for the intercept values?*

When intercept distribution data are plotted against intercept or area on a linear abscissa the distribution is compressed for the small sizes and expanded for the large sizes. This representation possibly reflects what is seen when the eye/brain interprets the microstructures with a bias towards the larger sizes. It enables differences to be seen more easily for large grains but not for the grains at the small end of the distribution. In conclusion, both plots could have a role to play depending on the kind of information required. Normalised abscissa values have some advantage when comparing materials with different mean values. It is possible to more clearly see differences in distribution widths.

Is there any benefit in plotting the ordinate on a linear scale rather than on probability paper?

The probability plots expand the distribution at each end and effectively weight the information so that perhaps an exaggerated effect of differences in large or small grains is observed.

- *Is there an advantage to plot the data as distance or area fractions (ie size fraction) rather than by number fraction? Is it number fractions or is it size fractions that are related to properties?*

It has been shown [2,3] that there are advantages to using areas rather than intercepts because the differences in distribution type for materials with narrow and wide distributions are more pronounced. Areas can be estimated by squaring the values of the intercepts (see section 5.1). Some typical plots of the different kinds of distributions are shown in Fig 4 using data from the NPL set of measurements on AA5182 and IN909. **Graphs of areas or estimated areas plotted against normalised size appear to give improved discrimination between materials with different grain size distributions.**

The issue of whether to use number or size probability and how they are calculated can be confusing when first addressed. Therefore a simple worked example will be included in the second part of this report [8] to help understand the concept.

5 MODELLING STUDIES

Computer simulation was used to assess the effectiveness of the linear intercept or area measurement techniques and its relevance to three dimensional structures [9,10]. A computer programme was written (called Polychop, a copy of which can be obtained from NPL) that will run on a standard PC. The program contains various shapes such as a tetrakaidecahedron, a cube, a cylinder, a rod, a tetrahedron and a trigonal prism. These shapes can be used to explore the consequent distribution of 2-D intercepts and areas that can be generated from the 3-D shape. The program generates statistical data on each shape for a specified number of sections. The data includes information on areas, intercept lengths, vertices and numbers of sides. The program can be run with single sizes of shapes or as normal (a Gaussian) or lognormal distributions (with different distribution widths and mean values). The data are generated by randomly sectioning the shape (distribution).

Three aspects of the measurement of distribution data using intercepts were investigated with the tetrakaidecahedron model shape (Fig 5):

- Estimating areas using squared values of intercepts
- Measurement of small intercepts
- Quantification of distribution width using model lognormal distributions.

5.1 AREA ESTIMATION

Direct measurement of the areas of grains is very time consuming without fully automated image analysis systems, especially as up to 200 grains probably need to be measured to obtain reasonable distribution data. However, it is possible to use the intercept measurements, by squaring the individual values, to obtain an estimate of area. This is not obviously reasonable because the probabilities of measurement associated with small intercepts through the edges of large grains and the probabilities of measurement related to the selection of small or large grains are not easy to quantify. Consequently, a tetrakaidecahedron model shape was used to directly compare data obtained by squaring intercepts and by direct area measurement. This data was obtained for three distributions of the tetrakaidecahedron shape.

- single sized
- lognormally distributed, $\sigma = 0.3$
- lognormally distributed, $\sigma = 0.6$

The results are shown in Fig 6 for size probabilities plotted against areas or estimated areas (intercept squared). The abscissa is plotted both directly and by normalising with the number average. It can be seen that the agreement between areas and estimated areas is very consistent. For the normalised plot there is a shift to higher values for the estimated area data but the trends (i.e. dependence on distribution width) are very similar. Clearly Fig 6 supports the use of intercept squared values as a measure of estimated area for investigating trends in distribution width.

5.2 SHORT INTERCEPTS

It is likely that in measurements on real microstructures that short intercept values are undercounted due to difficulties in image resolution and grain rounding. For this reason measurements at NPL from the structure of the Al alloy were compared with data from modelling a lognormally distributed (3-D) tetrakaidecahedron. The results are shown in Fig 7 plotted both as a number and a size probability. It can be seen that the number probability at low intercept values is much higher than the data from the model tetrakaidecahedron, as predicted. However, when the data are plotted as size probabilities this difference disappears, because clearly the fraction of area occupied by the small intercepts is tiny. Thus if size distributions are important then it is not necessary to measure the small intercepts with great accuracy.

5.3 DISTRIBUTION WIDTH

The tetrakaidecahedron was used to generate size probability data for three distributions:

- single sized
lognormally distributed, $\sigma = 0.3$
- lognormally distributed, $\sigma = 0.6$

The data are plotted in Fig 8 as cumulative size probability (estimated area - based on squaring the intercept values) against normalised intercept (ie intercepts normalised by the number mean linear intercept). This comparison plot can therefore be used to decide how uniform a particular structure is in 3-D. The plots are compared with measurements on the ILE alloy microstructures in section 6.4.

6 ILE RESULTS AND ANALYSIS

Participants provided the following results

- A calculated value of magnification.
- A calculated value of arithmetic mean linear intercept from the reference line.
- A calculated value of arithmetic mean linear intercept from their own set of lines on each image.
- A spreadsheet of individual intercept values for subsequent analysis.

The results were used to plot comparative figures of mean values and standard deviations together with analysis of different types of distribution data. An experimental investigation of the use of running average data values was also explored as a contribution towards answering the question - "how many intercept values need to be measured to give a specific value of uncertainty".

The distribution data were also compared with data from modelling studies using the tetrakaidecahedron shape.

6.1 MEAN VALUES

Mean values were obtained from 4 sets of measurements

- Magnification from reference line.
- Arithmetic mean linear intercept from the reference line.
- Arithmetic mean linear intercept from the participants lines.
- Mean value of estimated area from squaring the intercept data on participants lines.

Calculated magnifications from the single reference line and arithmetic mean linear intercepts and normalised values of these parameters are given in Table 1 and Figs 9-11. Arithmetic mean linear intercepts, arithmetic estimated areas and normalised values of these parameters from measurements on participants own lines are given in Table 2 and Figs 12-13. Values for the parameters and their coefficients of variation are summarised in Tables 3 and 4. Most partners measured magnification to within 0.5%.

Table 1
Reference Image - Single Line

AA5182	Calculated Magnification	Normalised Magnification	Arithmetic Mean Linear intercept, μm^*	Normalised intercept
NPL	370.9	1.0009	24.6	0.909
Alcan	371.0	1.0012	25.8	0.954
Rolls Royce	371.0	1.0012	25.0	0.925
Doncasters	370.9	1.0009	25.9	0.958
Firth Rixson	371.0	1.0012	24.9	0.922
Incotest	371.0	1.0012	24.7	0.913
LSM	370.0	0.9985	23.4	0.867
Sheffield Testing Laboratories	371.6	1.0028	24.7	0.913
LSM/PB	370.0	0.9985	24.3	0.899
LSM/SM	370.0	0.9985	25.4	0.940
Alstom	368.6	0.9947	25.3	0.936
Sheffield Hallam University	370.9	1.0009	24.6	0.980
Imaging Associates	370.4	0.9996	25.8	0.954

Waspaloy	Calculated Magnification	Normalised Magnification	Arithmetic Mean Linear intercept, μm^*	Normalised intercept
NPL	249.4	0.9993	28.3	0.911
Alcan	250.0	1.0017	29.3	0.941
Rolls Royce	249.4	0.9993	29.5	0.947
Doncasters	249.4	0.9993	29.0	0.932
Firth Rixson	249.4	0.9993	28.9	0.928
Incotest	249.0	0.9977	29.1	0.936
	250.0	1.0017	28.5	0.915
Sheffield Testing Laboratories	249.4	0.9993	29.1	0.936
LSM/PB	250.0	1.0017	27.5	0.884
LSM/SM	249.2	0.9985	27.9	0.895
Alstom	250.0	1.0017	30.6	0.984
Sheffield Hallam University	249.4	0.9993	29.0	1.010
Imaging Associates	250.0	1.0017	26.7	0.858

IN909	Calculated Magnification	Normalised Magnification	Arithmetic Mean Linear intercept, μm^*	Normalised intercept
NPL	493.8	0.9972	15.3	0.989
	495.0	0.9996	13.9	0.900
	493.8	0.9972	15.1	0.980
	496.3	1.0023	14.2	0.920
	495.0	0.9996	15.3	0.990
	494.0	0.9976	14.2	0.920
	495.0	0.9996	13.4	0.867
Sheffield Testing Laboratories	494.2	0.9980	14.1	0.913
LSM/PB	496.6	1.0029	13.5	0.871
	495.0	0.9996	13.6	0.881
Alstom	494.6	0.9988	14.9	0.965
Sheffield Hallam University	494.0	0.9976	15.1	1.070
Imaging Associates	500.0	1.0097	12.7	0.823

* About 20 intercepts were measured along the reference line

Table 2
Reference Image - Participants Lines

AA5182	Calculated Magnification	Arithmetic Mean Estimated Area, μm^2	Arithmetic Mean Linear intercept, μm	Normalised intercept	Number of intercepts measured
NPL	370.9	1091	28.0	1.085	112
Alcan	371.0	1047	27.4	1.061	54
Rolls Royce	371.0	978	26.6	1.030	79
Doncasters	370.9	795	25.9	1.003	58
Firth Rixson	371.0	1199	26.3	1.019	41
Incotest	371.0	999	25.5	0.988	41
LSM	370.0	1012	22.9	0.887	36
Sheffield Testing Laboratories	371.6	733	26.0	1.007	91
LSM/PB	370.0	748	22.9	0.887	41
LSM/SM	370.0	1014	26.5	1.027	38
Alstom	368.6	1060	28.2	1.092	36
Sheffield Hallam University	370.9	816	23.6	0.914	59
Imaging Associates	370.4	882	25.8	0.999	79

Waspaloy	Calculated Magnification	Arithmetic Mean Estimated Area, μm^2	Arithmetic Mean Linear intercept, μm	Normalised intercept	Number of intercepts measured
NPL	249.4	807	21.6	0.794	182
Alcan	250.0	1357	27.6	1.015	86
Rolls Royce	249.4	1801	32.0	1.177	76
Doncasters	249.4	1631	31.3	1.151	76
Firth Rixson	249.4	1624	30.8	1.133	51
Incotest	249.0	958	24.8	0.912	63
LSM	250.0	1126	31.5	1.158	62
Sheffield Testing Laboratories	249.4	1639	24.7	0.908	100
LSM/PB	250.0	1297	25.4	0.934	65
LSM/SM	249.2	636	20.2	0.743	72
Alstom	250.0	1456	29.5	1.085	48
Sheffield Hallam University	249.4	1388	28.0	1.030	77
Imaging Associates	250.0	1248	26.1	0.960	113

IN909	Calculated Magnification	Arithmetic Mean Estimated Area, μm^2	Arithmetic Mean Linear intercept, μm	Normalised intercept	Number of intercepts measured
NPL	493.8	374	13.5	1.053	150
Alcan	495.0	301	13.0	1.014	93
Rolls Royce	493.8	352	12.9	1.007	106
Doncasters	496.3	304	13.1	1.022	90
Firth Rixson	495.0	269	12.0	0.936	63
Incotest	494.0	243	11.8	0.921	70
LSM	495.0	253	12.8	0.999	64
Sheffield Testing Laboratories	494.2	264	12.5	0.975	125
LSM/PB	496.6	294	12.5	0.975	64
LSM/SM	495.0	396	13.4	1.046	60
Alstom	494.6	332	13.0	1.014	60
Sheffield Hallam University	494.0	335	13.7	1.069	84
Imaging Associates	500.0	306	12.4	0.968	119

Table 3 - Mean Values

	Magnification	Mean linear intercept (reference line) μm	Mean linear intercept (from participants line) μm	Mean Area* (Estimated) μm^2
AA5182	370.6	25.0	25.8	952
Waspalloy	249.6	28.7	27.2	1305
IN909	495.2	14.3	12.8	310

*Estimated from squaring intercept values.

**Table 4 - Summary of Measurements
Coefficient of Variation, %**

	Magnification	Mean linear intercept (from reference line)	Mean linear intercept (from participants line)	Mean Area (Estimated) μm^2
AA5182	0.20	2.7	6.5	14.6
Waspalloy	0.14	3.3	13.5	25.6
IN909	0.33	5.6	4.2	14.6

6.2 DISTRIBUTION PARAMETERS

Following the discussion in section 4 the data obtained from the ILE was analysed and plotted in graphical form using the following methods:

Set A	Number probability against Intercept	Fig 15
Set B	Area (E)* probability against Area (E)*	Fig 16
Set C	Area (E)* probability against Normalised Intercept**	Fig 17

*Area (E) is estimated area from squaring intercept values.

**Intercept values normalised by the arithmetic mean.

Graphs are shown in Figs 15-17. Each figure comprises a set of graphs from the individual laboratory participants, with the materials plotted on the same graph for comparison. All the data was plotted as cumulative distributions - this avoids arbitrary decisions about classifying intercept values in bins. The small number of measurements obtained by each participant on these reference images was also not helpful if classification into bins was required.

Set A - The number probability plots against intercept value all have a characteristic Γ shape, with a steep initial portion and large tail. The length of the tail corresponds to the larger grains and is more extensive in those alloys with a wider distribution of grains. The plot is not helpful in comparing distribution widths because each alloy has a different mean value of sizes. There is also considerable scatter in the data from organisation to organisation because of the small number of intercepts measured.

Sets B and C - The use of area probability (estimated) plots converts the Γ shape of the number probability plots to a fan-shape with the material with the more uniform distribution having a distribution plot closer to the vertical than the plots of materials with wider size distributions. This is consistent with the modelling predictions (see section 5). Also it is easier to compare the absolute width of the distributions using this type of plot when the abscissa is normalised (Fig 17). The separation between the materials is particularly clear to see in the NPL data where more intercepts were counted.

Scatter

Plots of area probability against normalised intercept are combined for each material in Fig 18 to show the scatter in distribution. The width of the band is an indication of the scatter. This is of greater extent in the materials with a wider grain size distribution. This is perhaps to be expected when in many cases less than 100 intercepts were measured.

6.3 RUNNING AVERAGE ANALYSIS

In order to provide information to address the question "how many intercepts need to be counted to give a specified degree of accuracy" the intercept data was plotted from each participant as a running average mean. This was performed for both intercept and estimated area (intercept squared) values:

Set D Running average intercept against number

Set E Running average area (E) against number

The results are shown in Figs 19 and 20.

In most cases it can be seen that in both types of graph the running average eventually settles down to a steady value. This happened more quickly on the intercept data than on the estimated area data and more quickly on the Al alloy than the two wide grained Ni alloys. However, there was considerable variation in the final values, as might be expected from the low numbers of intercepts counted in this exercise. In order to normalise the data plots were produced of the % deviation of each running average value from the final value. Horizontal lines were drawn on each of these plots corresponding to deviations of 20, 10, 5 and 2% from the final value. The number of intercepts associated with each of these deviations was then plotted against the deviation for each participant. These plots are shown in Fig 21 for each of the materials and each participant individually, and averaged in Fig 22. The mean number required for a smaller percentage deviation increases progressively for all the materials and is greater for the wide grained Ni alloys than for the more uniform Al alloy.

6.4 COMPARISON WITH MODELS

Both number and size probability distributions were generated from the ILE intercept data measured at NPL and compared with the tetrakaidecahedron models for both the AA5182 (Al alloy) and the Waspaloy (Ni alloy) materials.

The data are shown plotted in Figs 23-24 for different types of distribution:

Number probability:	Normalised intercept
Number probability:	Normalised estimated area (squared intercepts)
Size (length) probability:	Normalised intercept
Size (length) probability:	Normalised estimated area (squared intercepts)
Size (estimated area) probability:	Normalised intercept
Size (estimated area) probability:	Normalised estimated area (squared intercepts)

It is concluded that the most convenient distribution by which to discriminate the distribution widths of the real microstructures was that based on:

Estimated area probability:	Normalised linear intercept
-----------------------------	-----------------------------

It is also clear that the size distribution data from the Al alloy best fits the model (tetrakaidecahedron) data for the lognormal distribution with a σ of 0.3 while the size distribution data for the Ni alloy (Waspaloy) best fits the tetrakaidecahedron model data with a distribution width, σ of 0.6.

7 SUMMARY

Grain size distribution data have been obtained, through an interlaboratory intercomparison, on reference images prepared at NPL from three materials:

- Al alloy - AA5182
- Ni alloy - Waspaloy
- Ni alloy - IN 909

The Al alloy contained a nominally uniform grain size distribution while the two Ni alloys contained mixed grain sizes.

Data from the reference images were compared with data generated from a 3-D model shape, a tetrakaidecahedron. Analysis of the model data indicated that the use of size distributions rather than number distributions enabled different structures to be discriminated more easily with respect to estimating the width of the distribution. However, this conclusion was reached on limited data because only one image from each material was measured. The size distribution approach will be evaluated more carefully in part II of this report which will contain measurement data from more images/fields.

The measurements in this report therefore describe how well the data characterises these reference images, not how well the "material microstructures" are quantified.

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9 ACKNOWLEDGEMENTS

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LIST OF CAPTIONS

	Al alloy - AA5182.
	Ni alloy - Waspaloy.
	Ni alloy - IN909.
	Different distribution types.
Fig 5	Tetrakaidecahedron model shape.
	Comparison of direct area measurements with squared intercept values from the tetrakaidecahedron model.
	Comparison of number and size distributions for small intercept values.
Fig 8	Cumulative size probability data from tetrakaidecahedron models with different distribution widths.
	Normalised values of calculated magnifications.
Fig 10	Arithmetic mean linear intercepts from the reference line.
Fig	Normalised values of mean linear intercept from the reference line.
	Arithmetic mean linear intercepts from participants lines.
Fig 13	Normalised values of mean linear intercept from participants lines.
	Normalised values of mean estimated area from participants lines.
Fig 15	Number probability plotted against intercept.
	Area (E) probability plotted against Area (E).
	Area (E) probability plotted against Normalised Intercept.
Fig 18	Area (E) probability plotted against Normalised Intercept - all participants for each material.
	Running average intercept plotted against Number.
Fig 20	Running average area (E) plotted against Number.
Fig 21	Percentage deviation from running average plotted against Number for each participant.
	Mean percentage deviation from running average plotted against Number.
	Comparison of distribution data from AA5182 and tetrakaidecahedron model.
	Comparison of distribution data from Waspaloy and tetrakaidecahedron model.

image 9, AA5182, Macroviewer

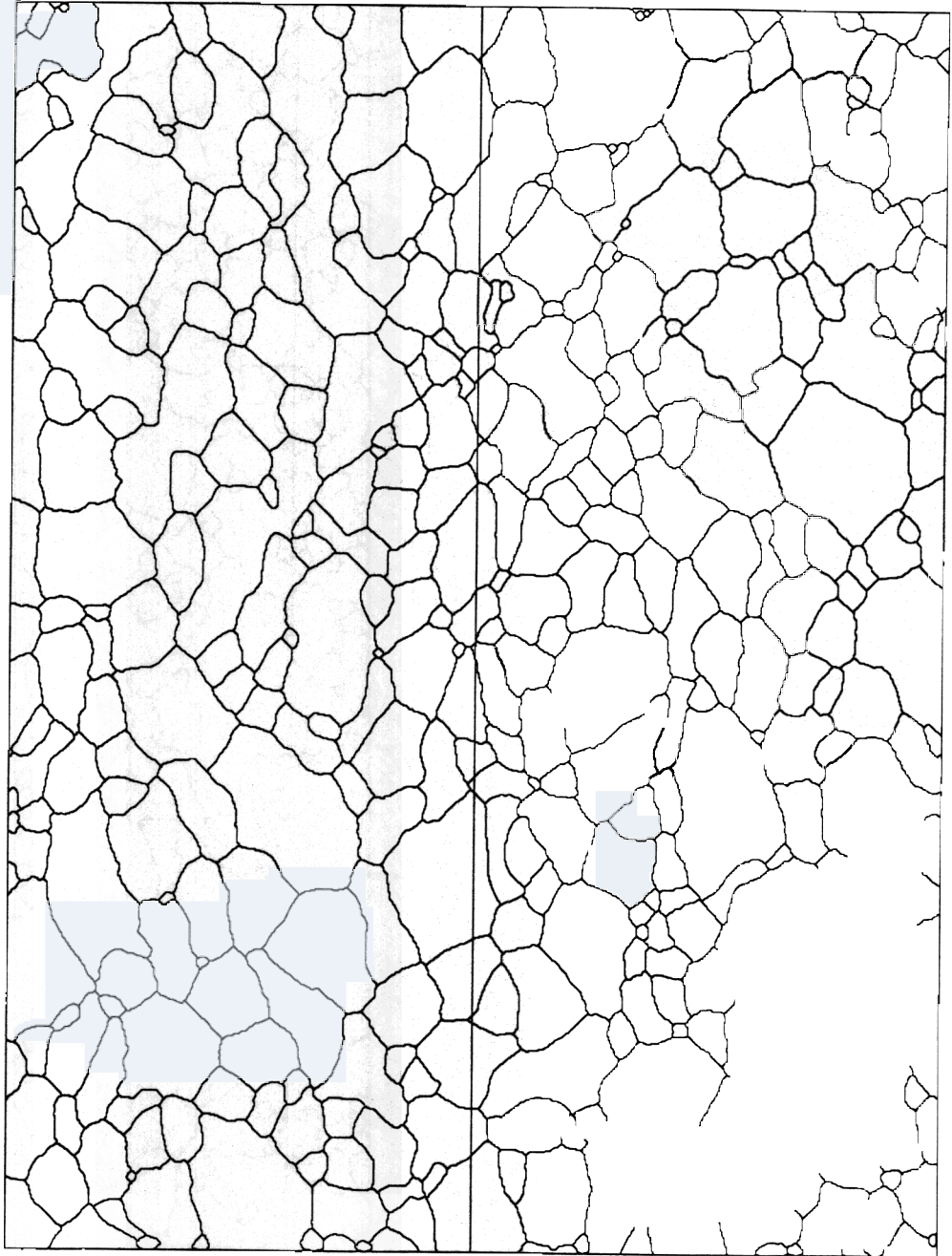


Fig 1

Al alloy - AA5182.

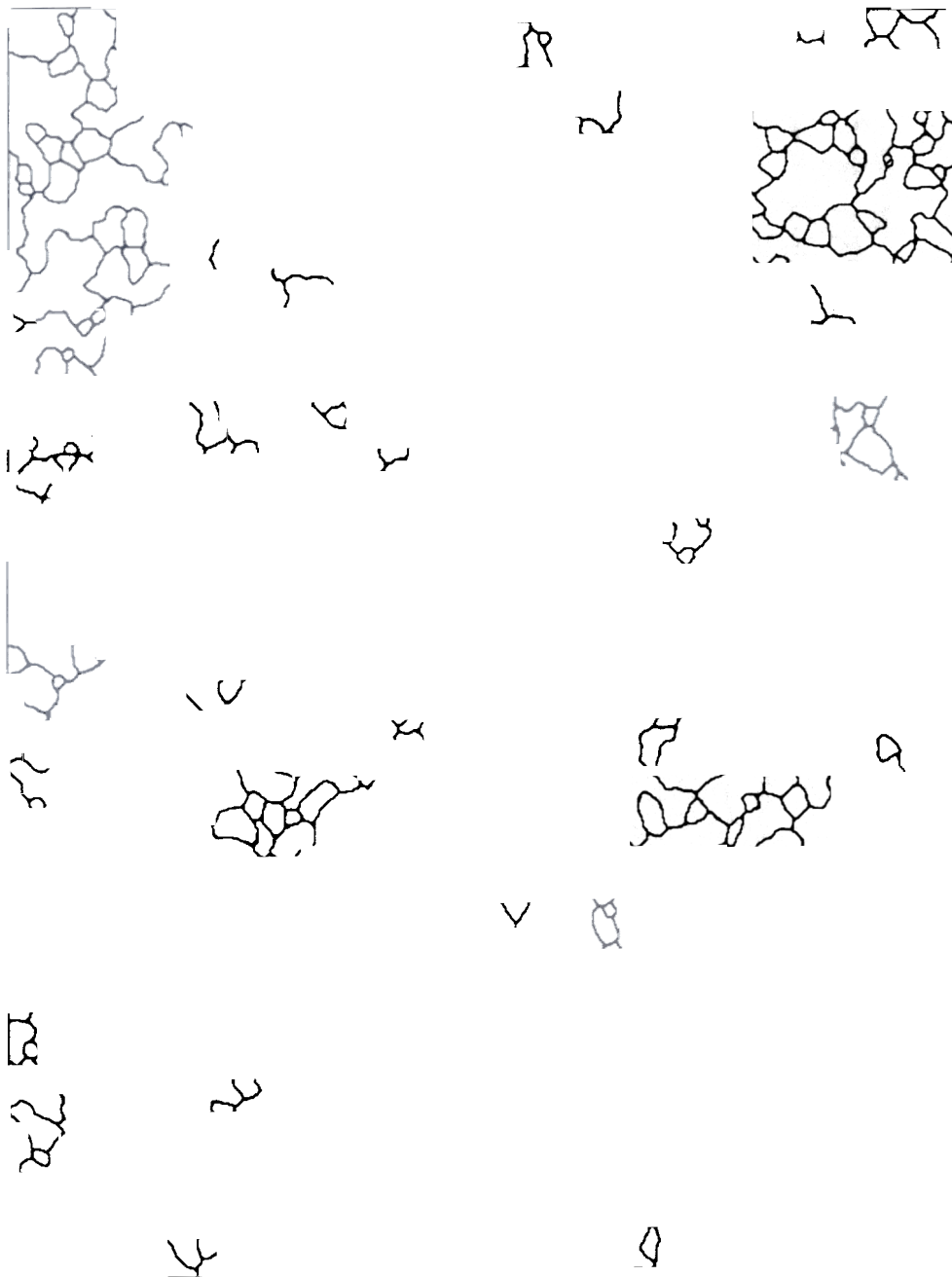


Fig 2 Ni alloy - Waspaloy.

mage 6, IN909, x20 objective



Fig 3

Ni alloy - IN909

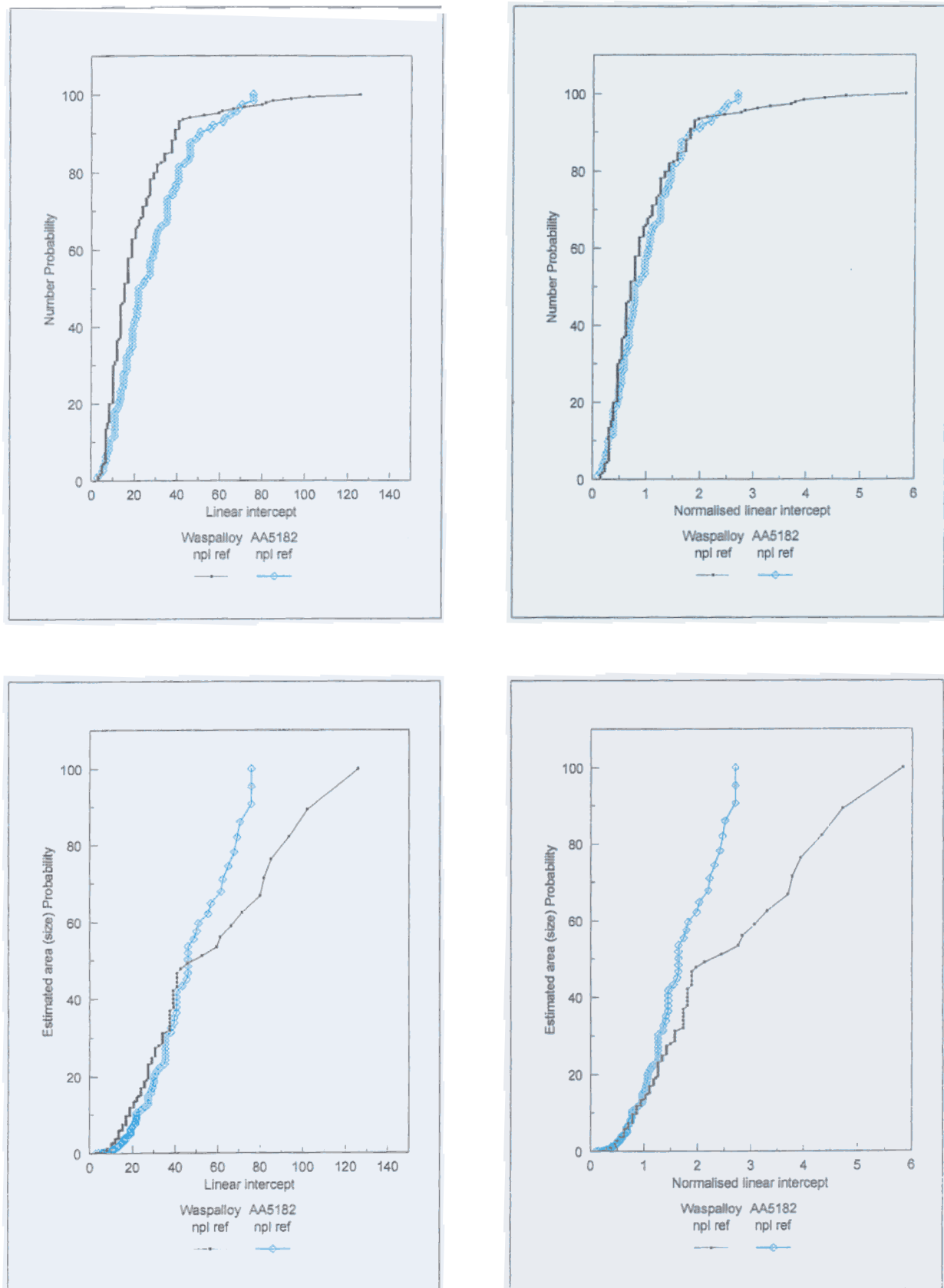


Fig 4 Different distribution types.

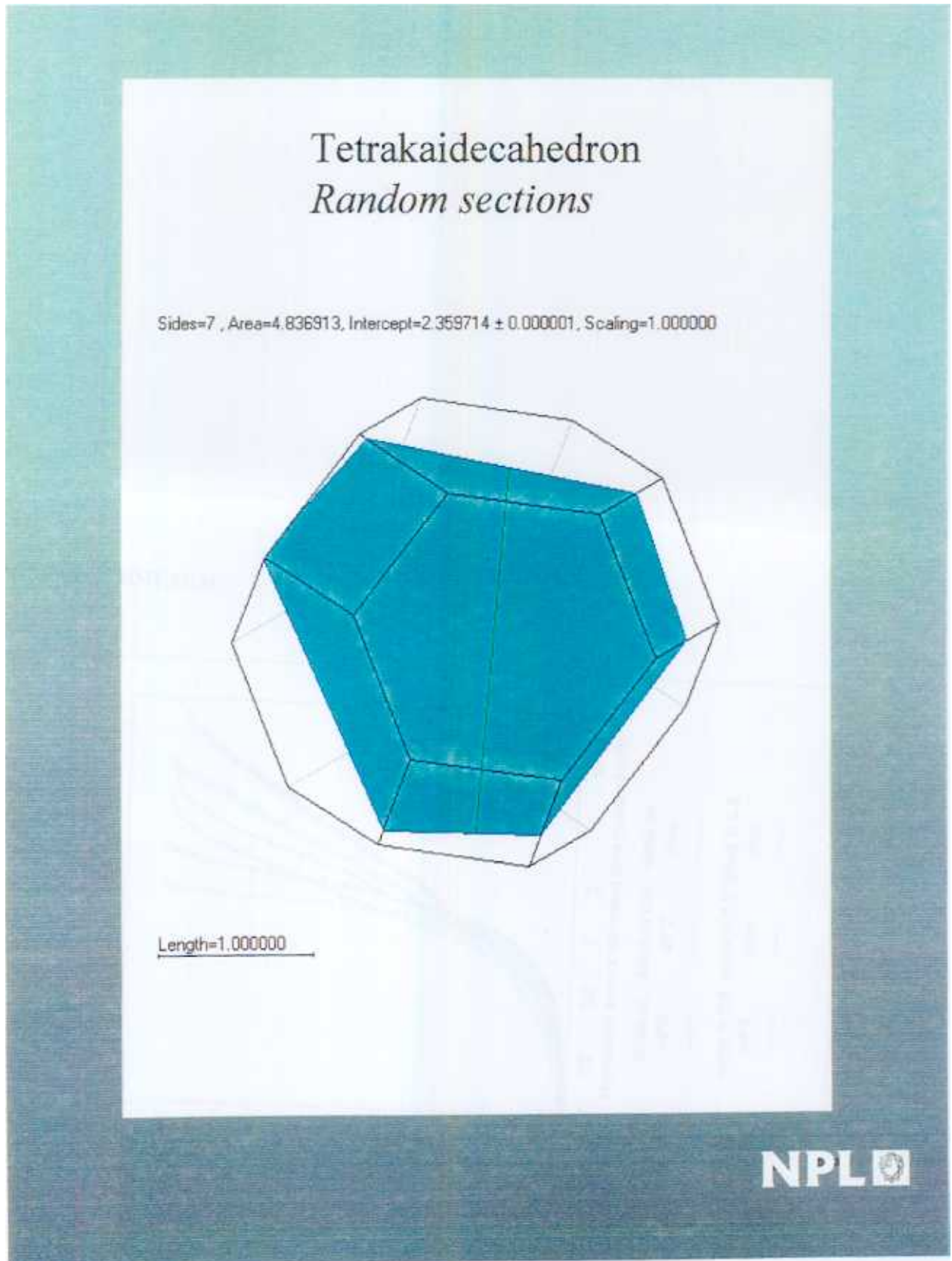


Fig 5 Tetrakaidecahedron model shape.

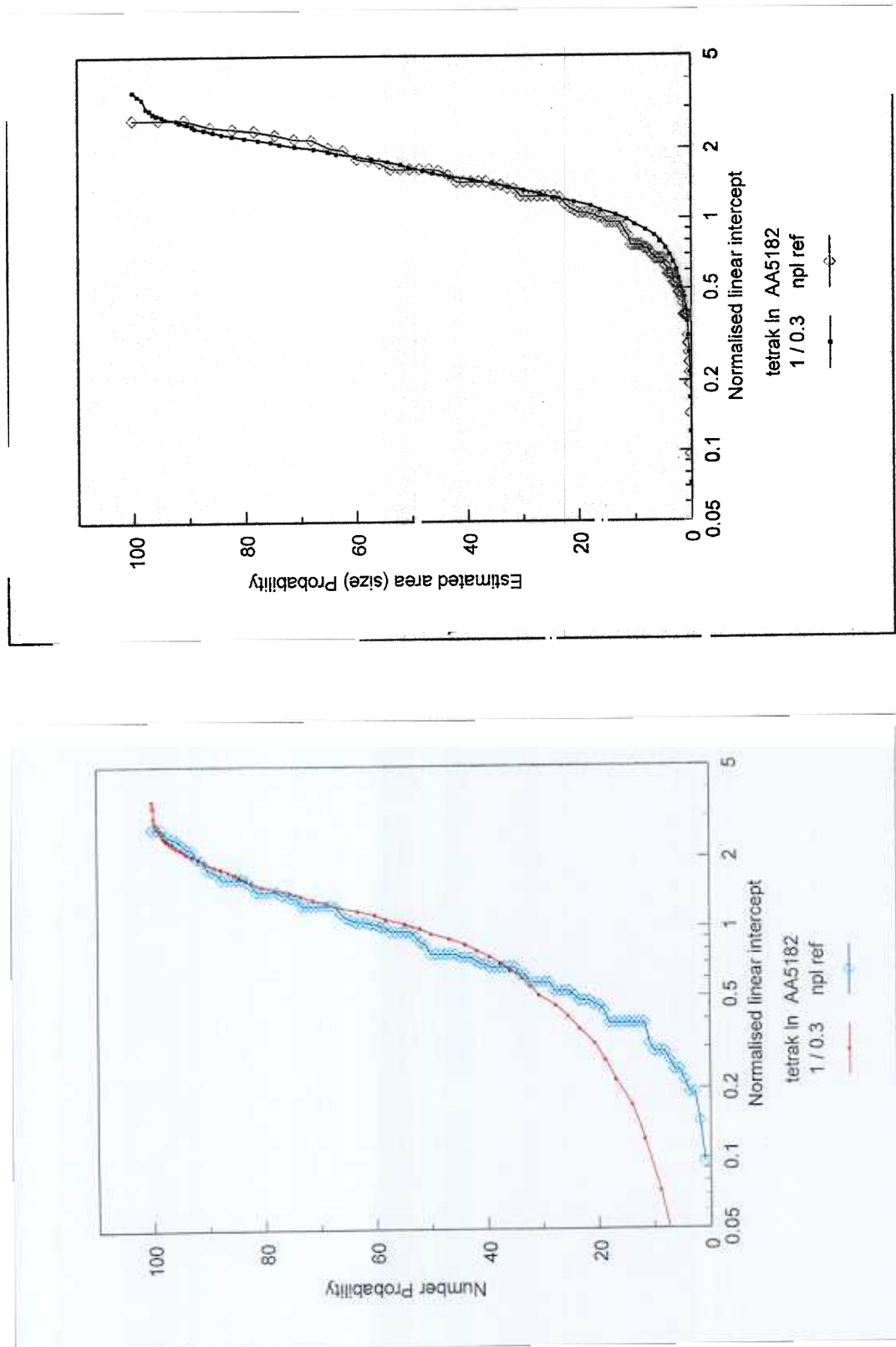


Fig 7

Comparison of number and size distributions for small intercept values.

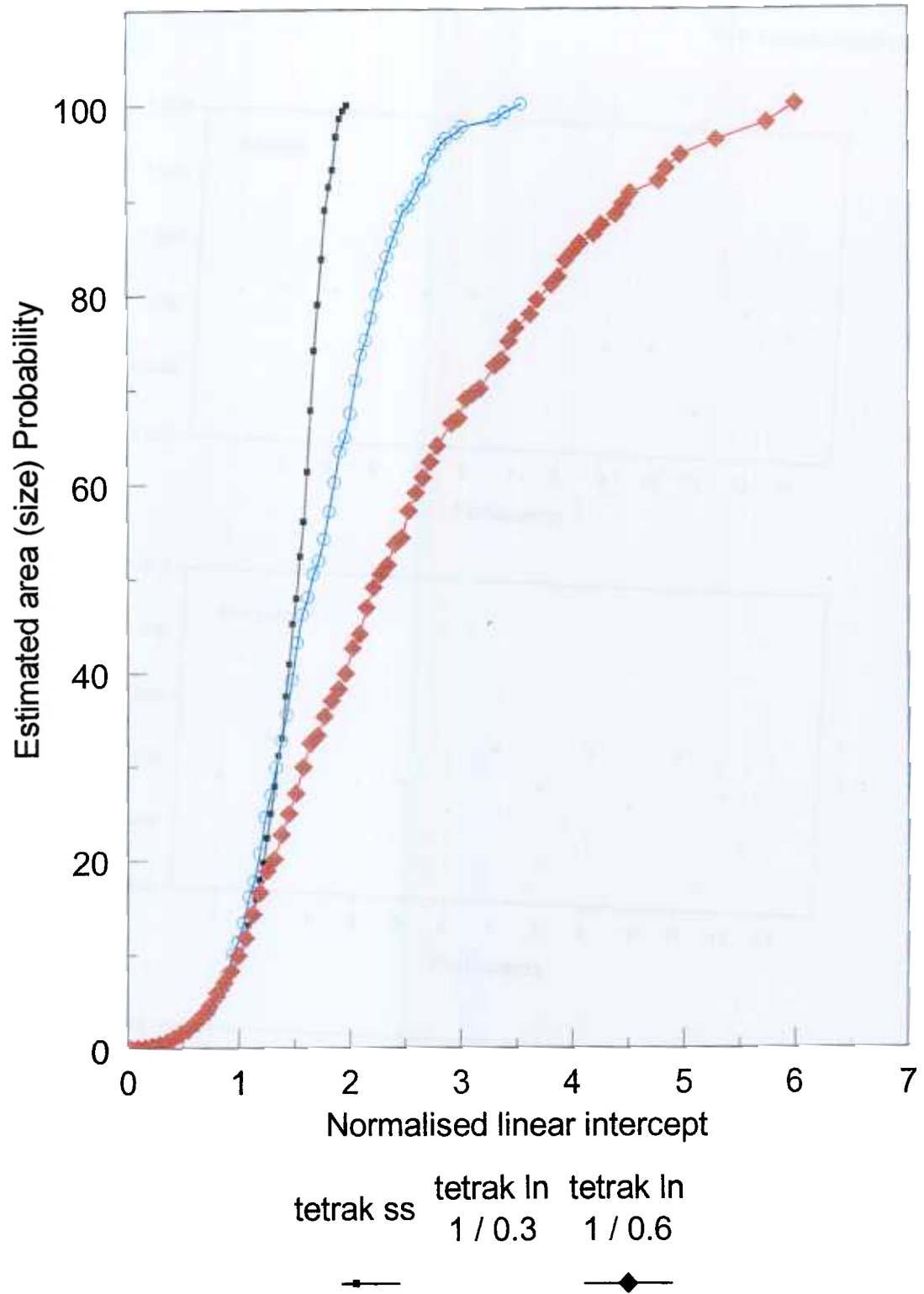


Fig 8 Cumulative size probability data from tetrakaidecahedron models with different distribution widths.

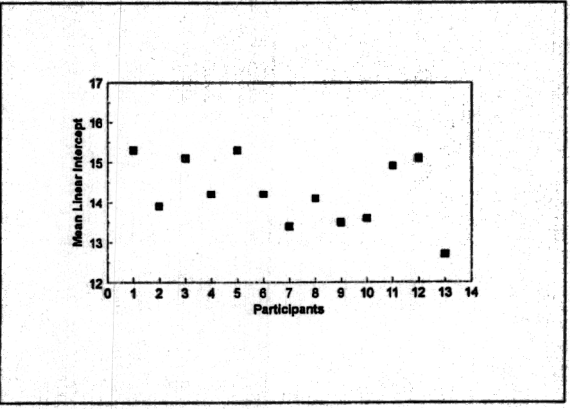
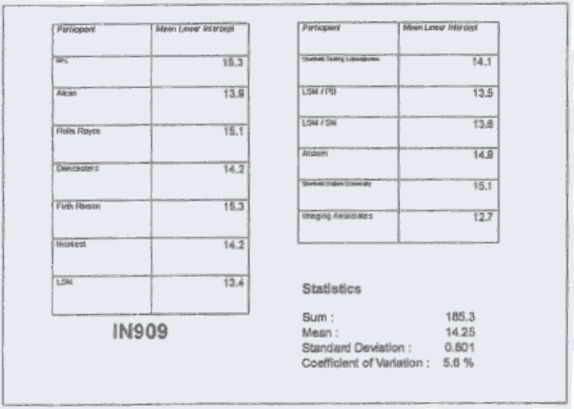
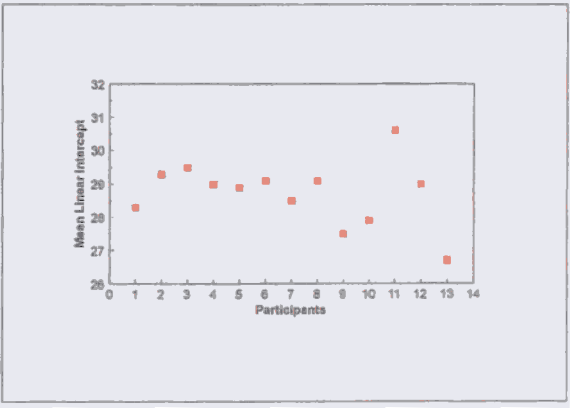
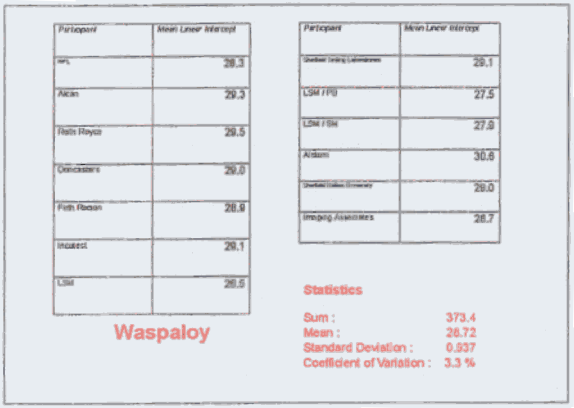
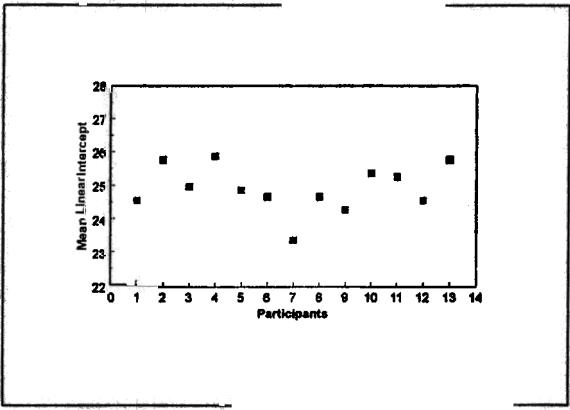
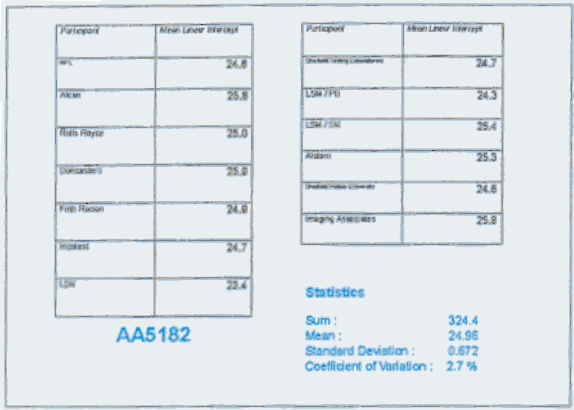


Fig 10 Arithmetic mean linear intercepts from the reference line.

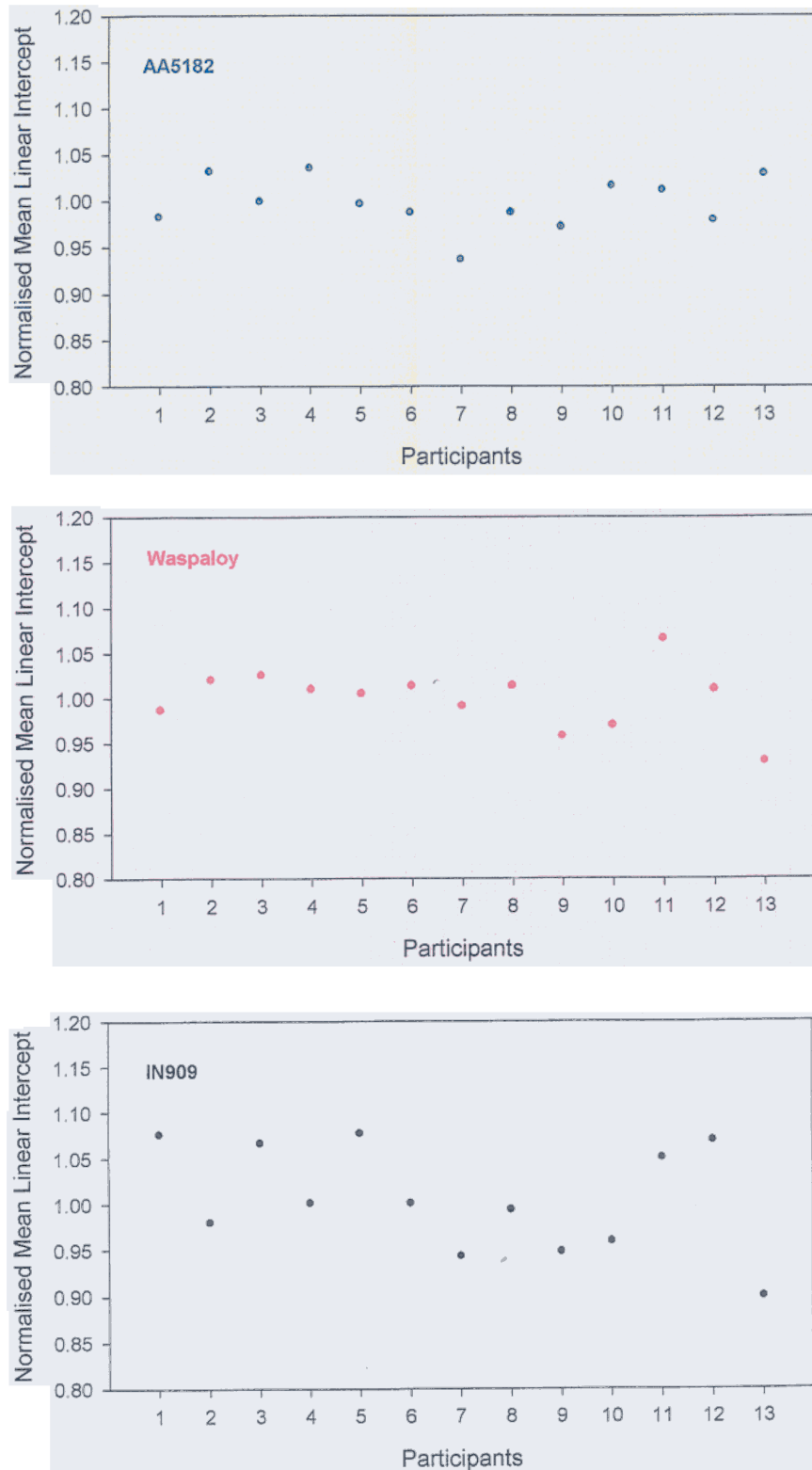


Fig 11 Normalised values of mean linear intercept from the reference line.

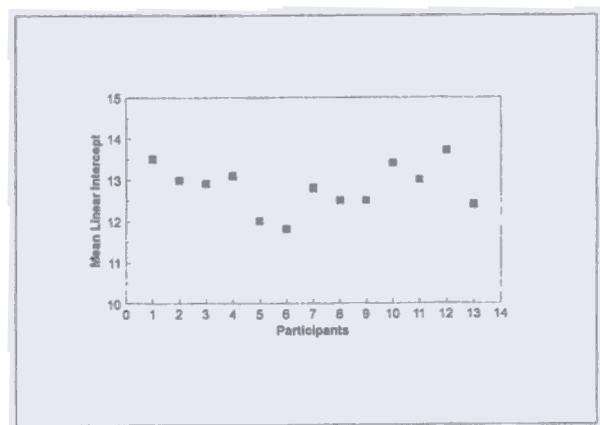
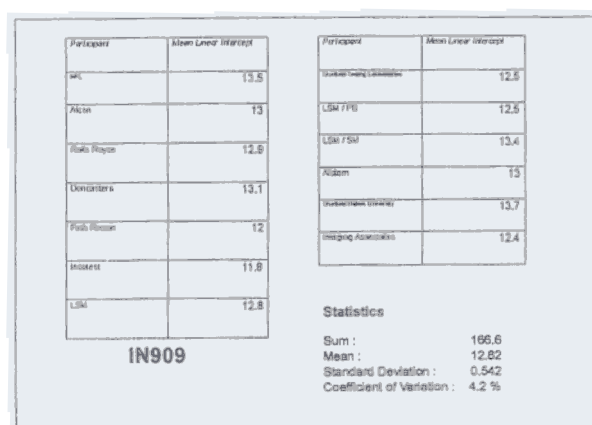
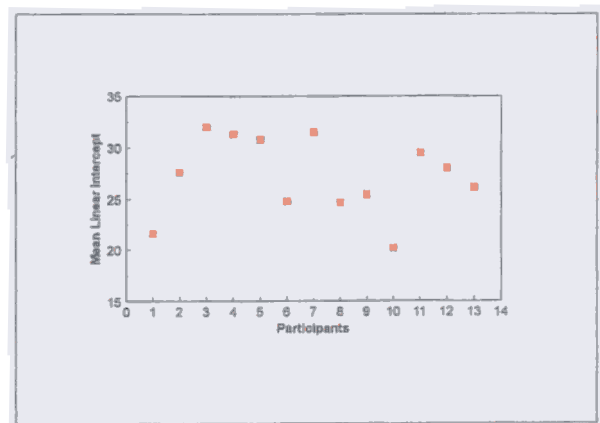
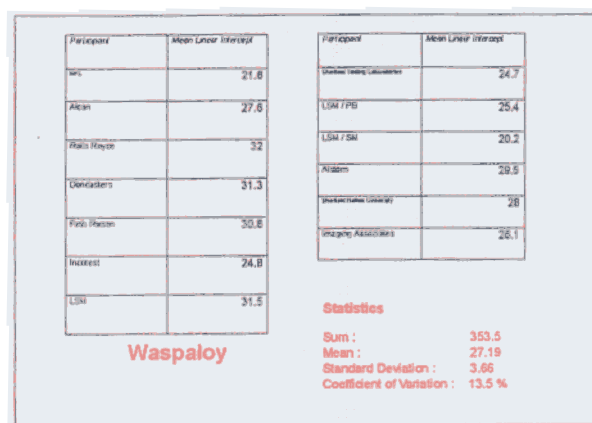
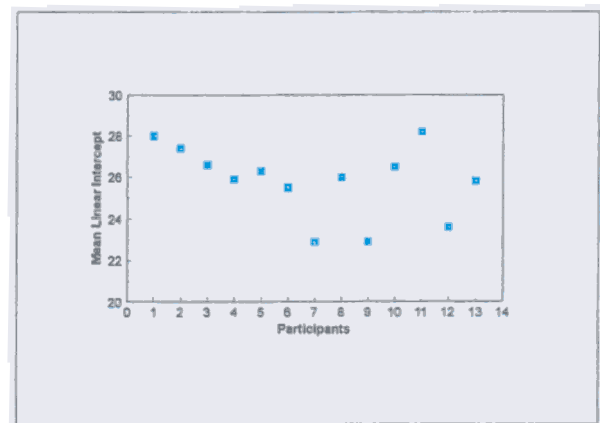
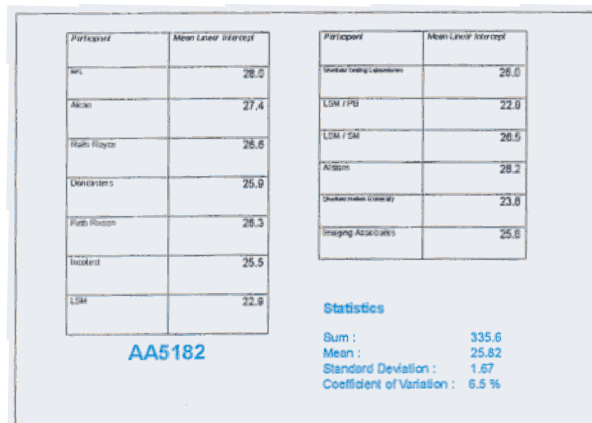


Fig 12 Arithmetic mean linear intercepts from participants lines.

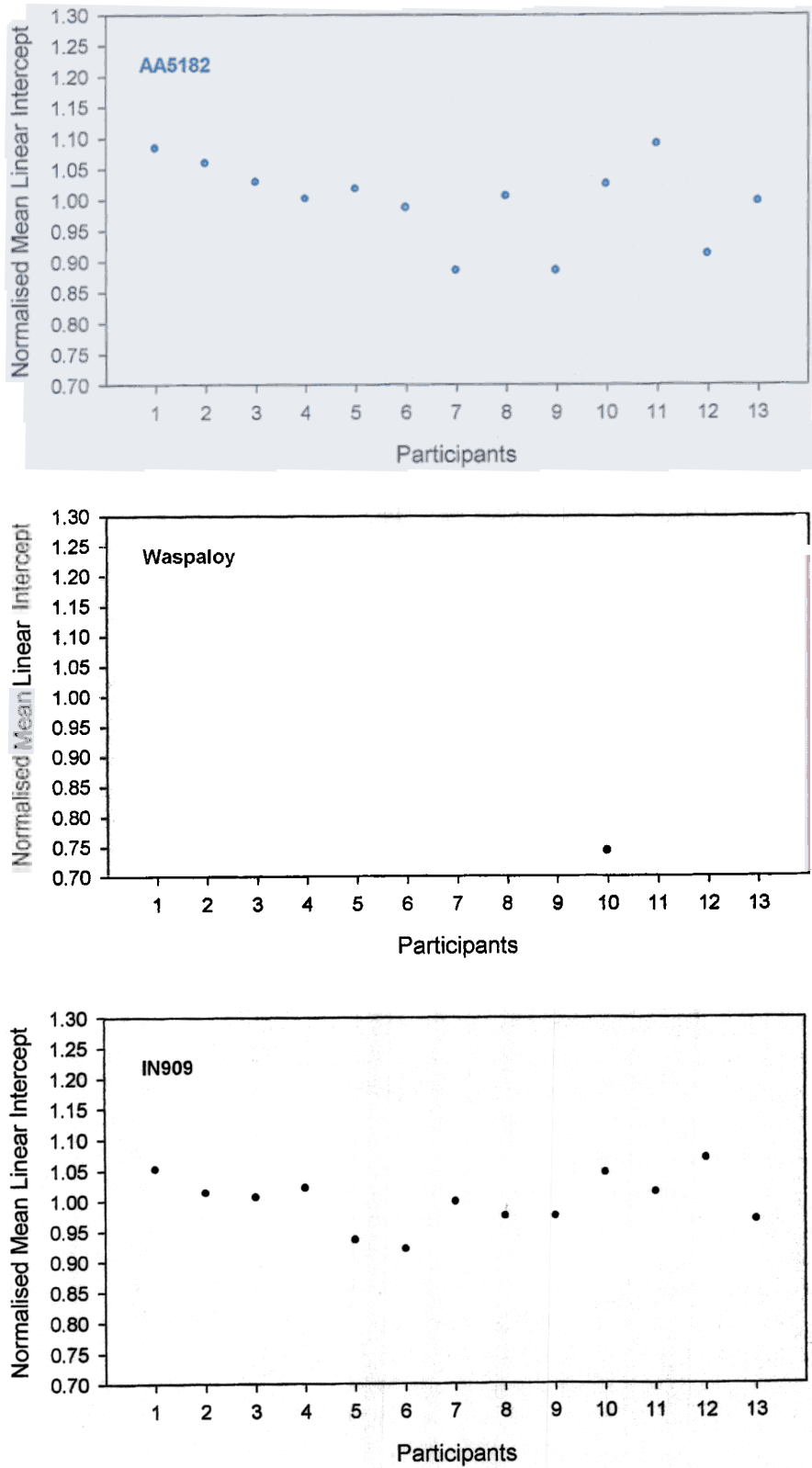


Fig 13 Normalised values of mean linear intercept from participants lines

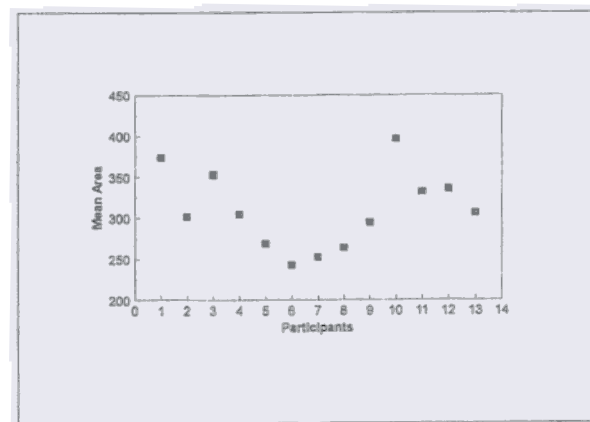
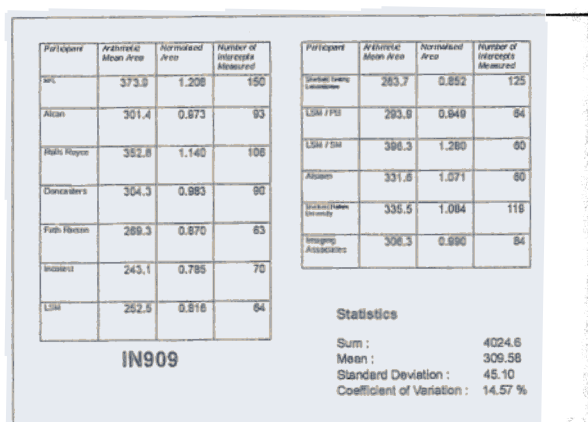
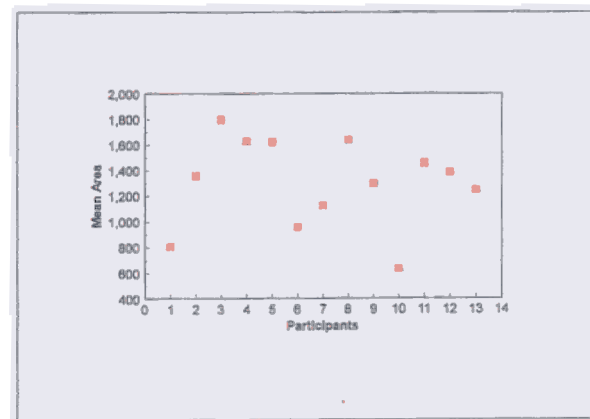
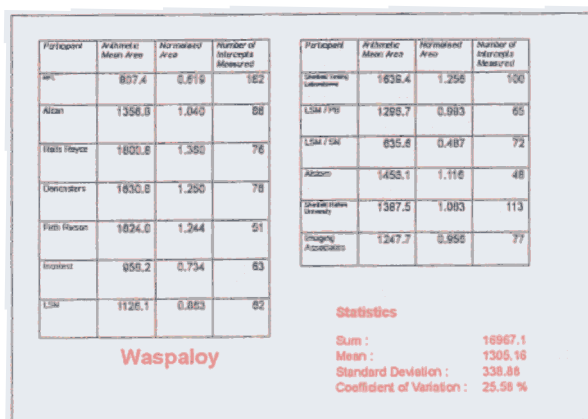
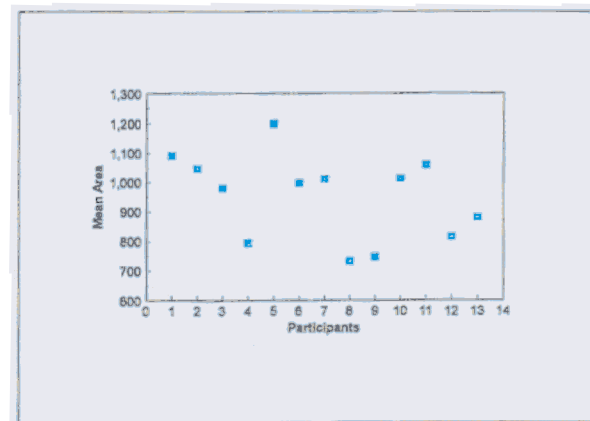
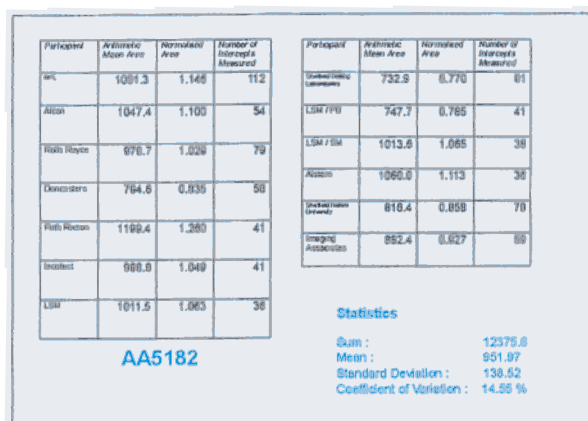
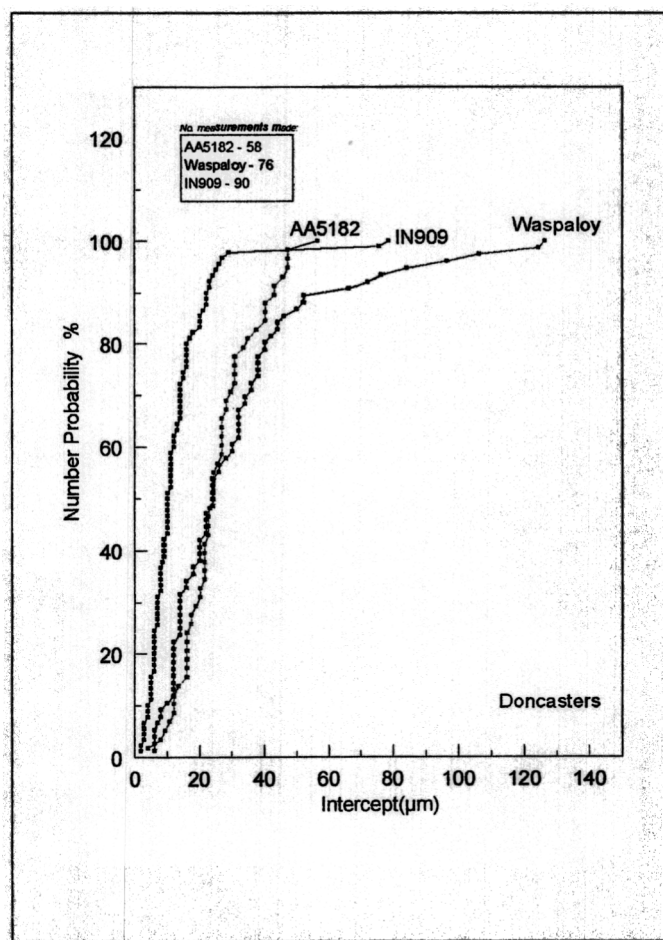
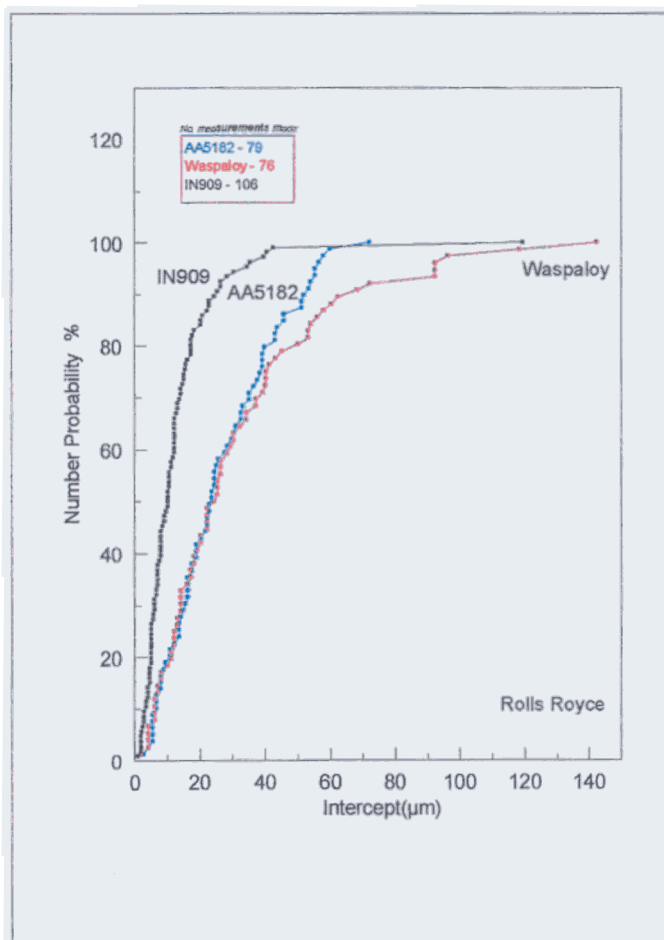
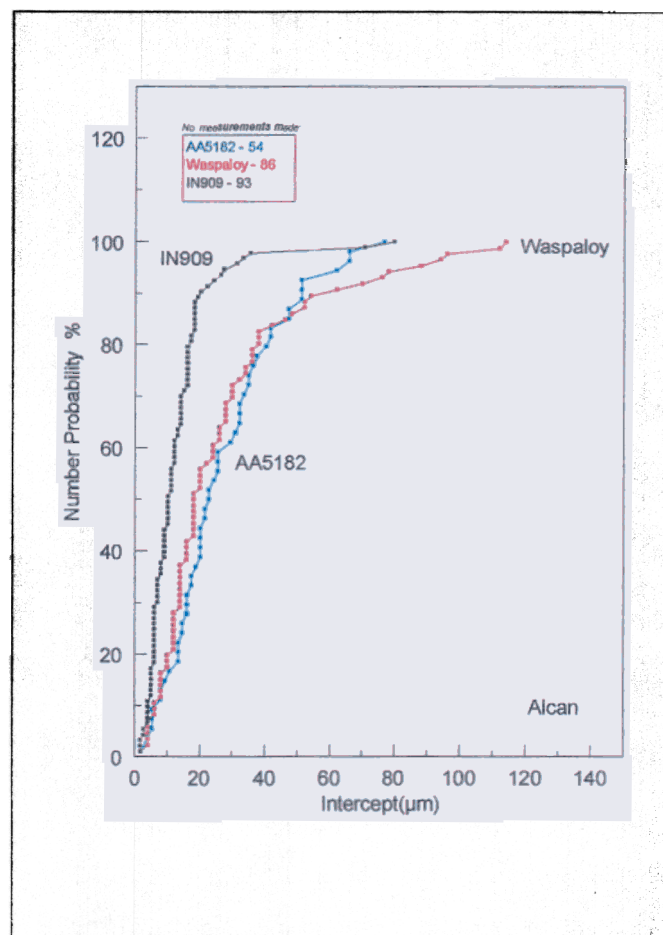
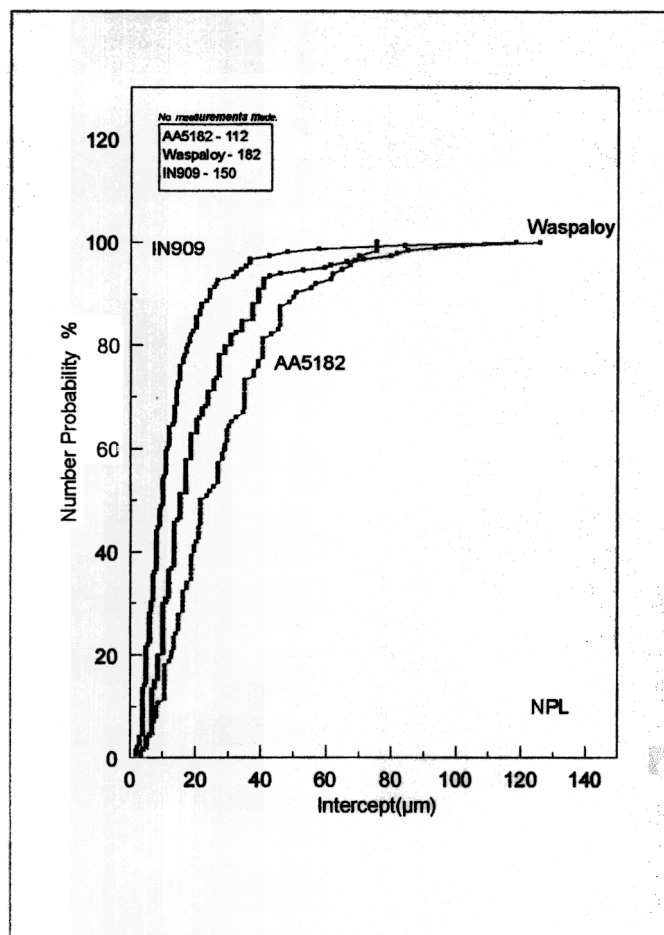
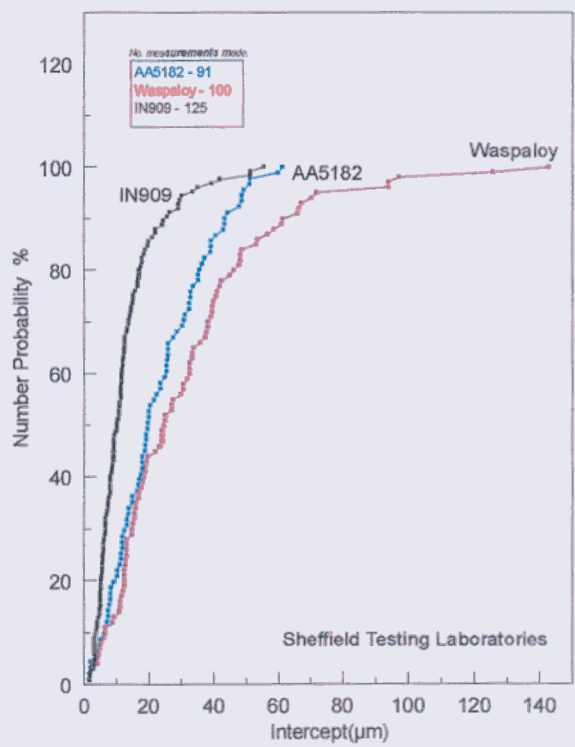
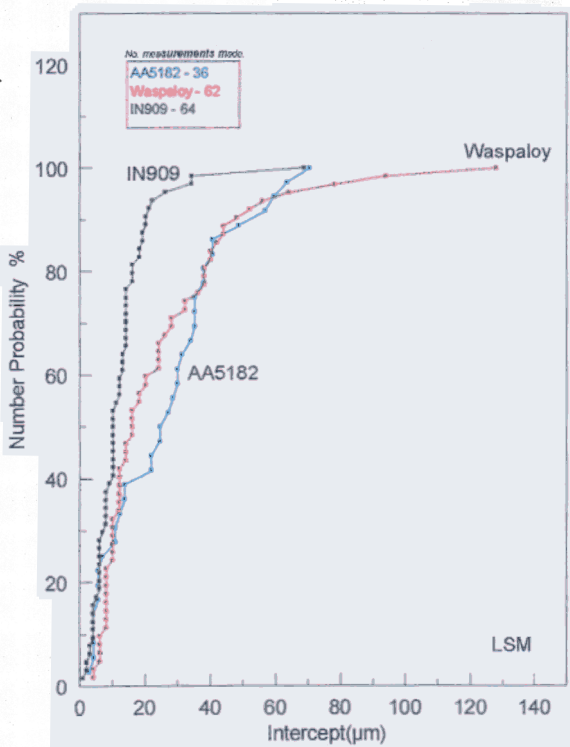
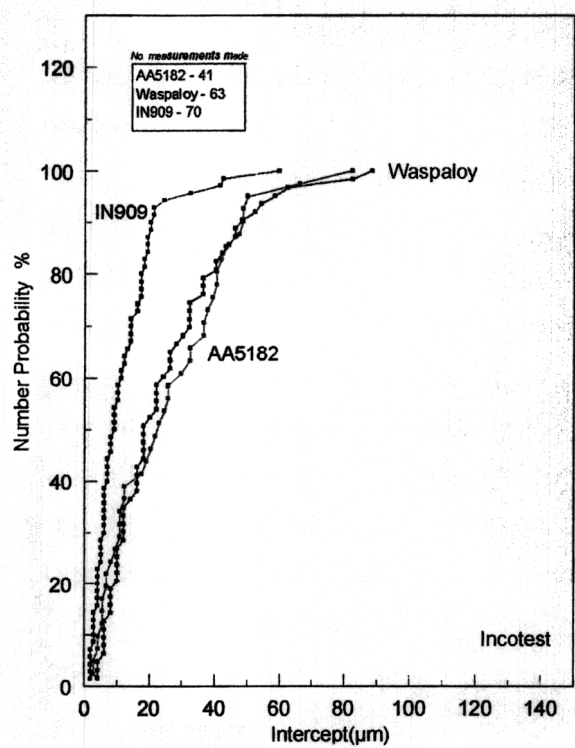
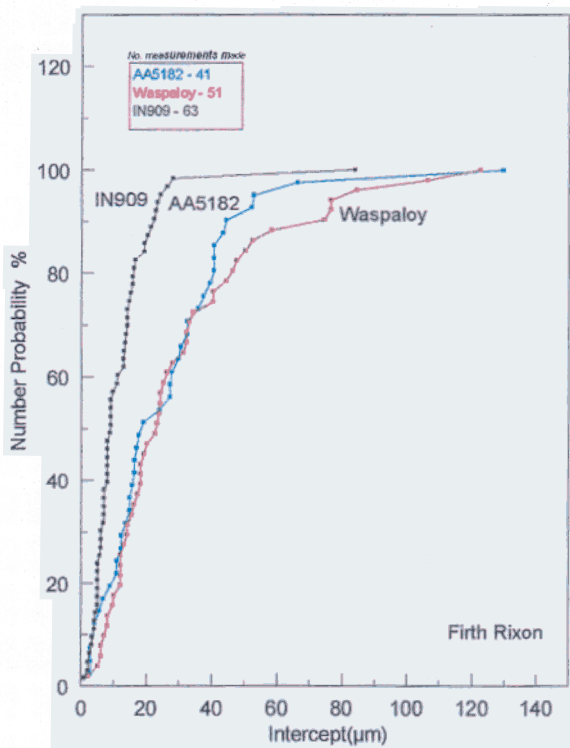
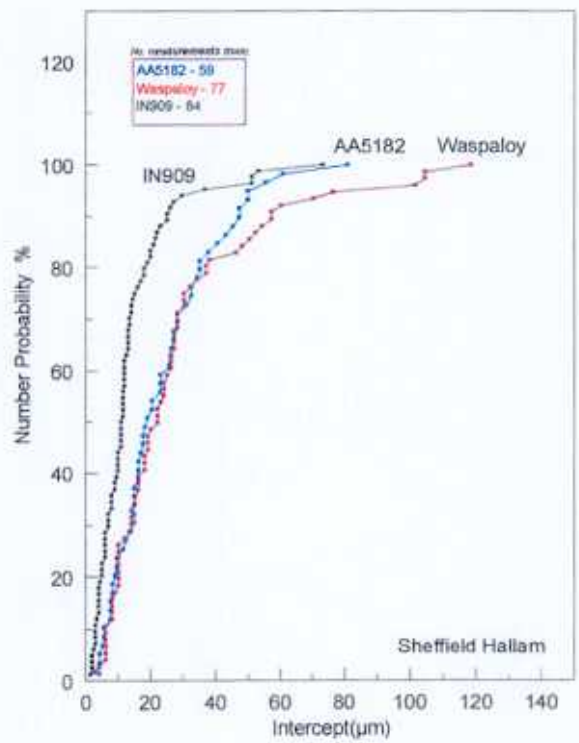
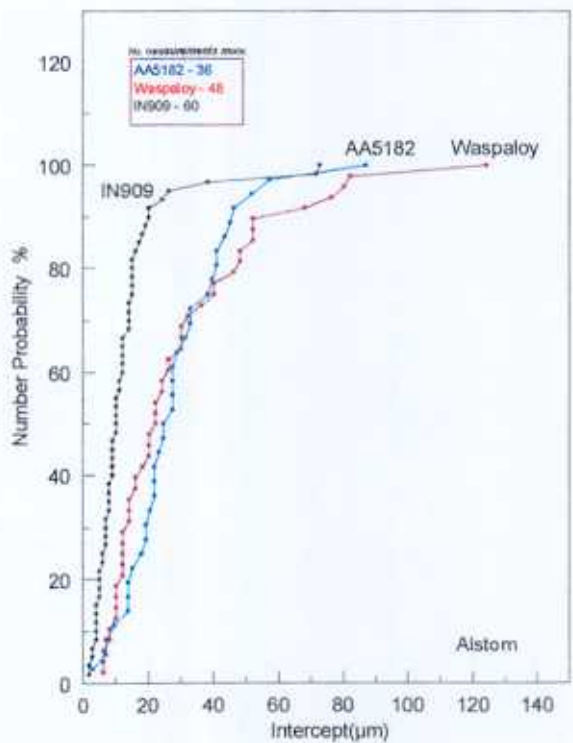
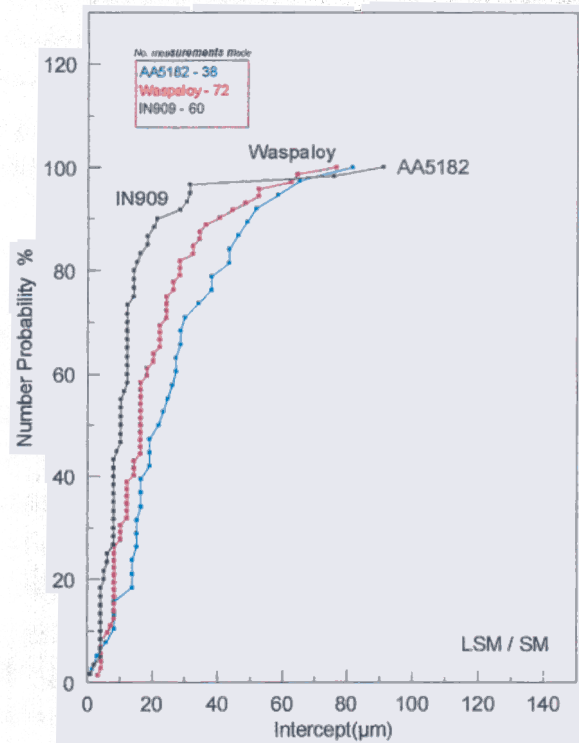
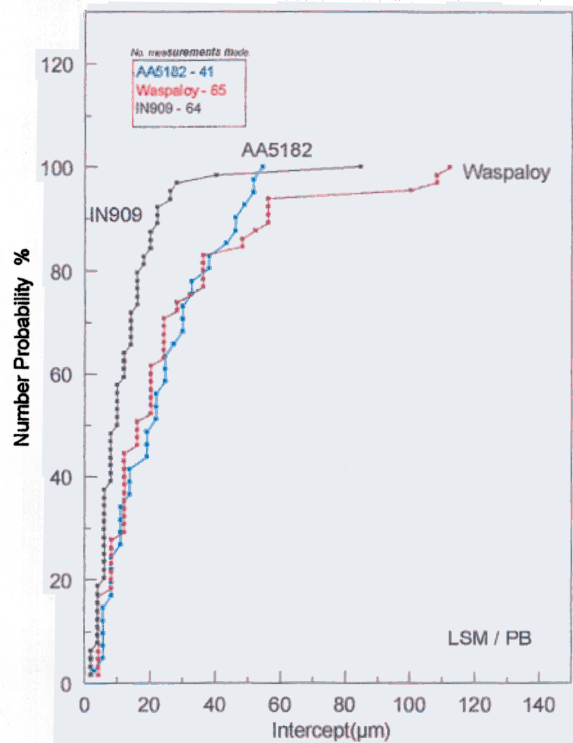


Fig 14 Normalised values of mean estimated area from participants lines.

Fig 15 Number probability plotted against intercept.







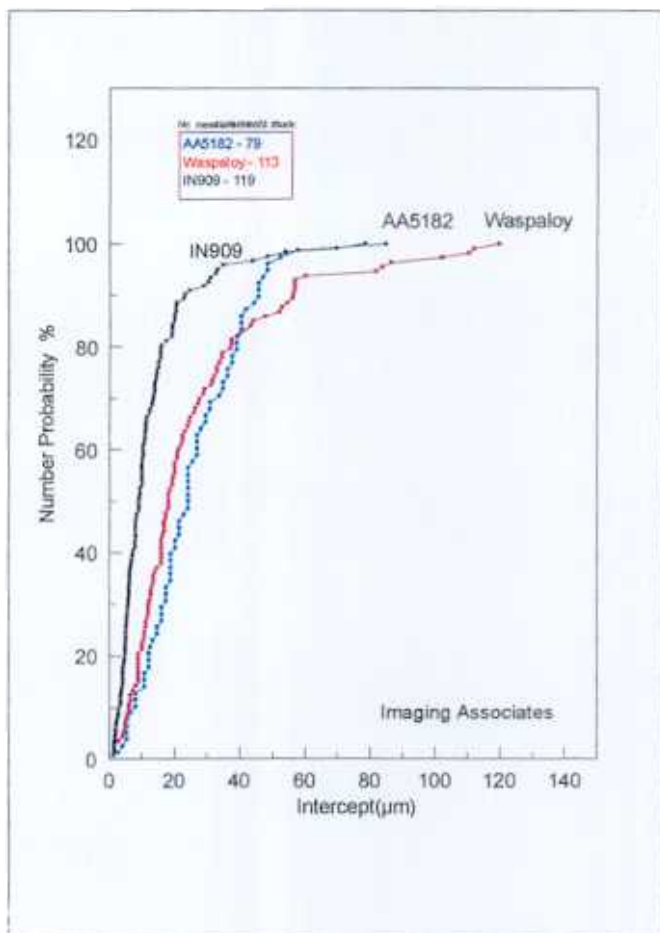
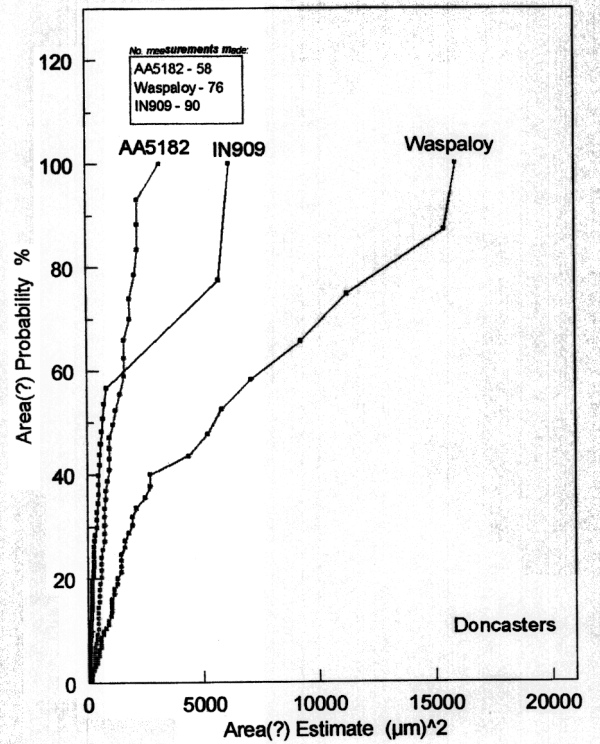
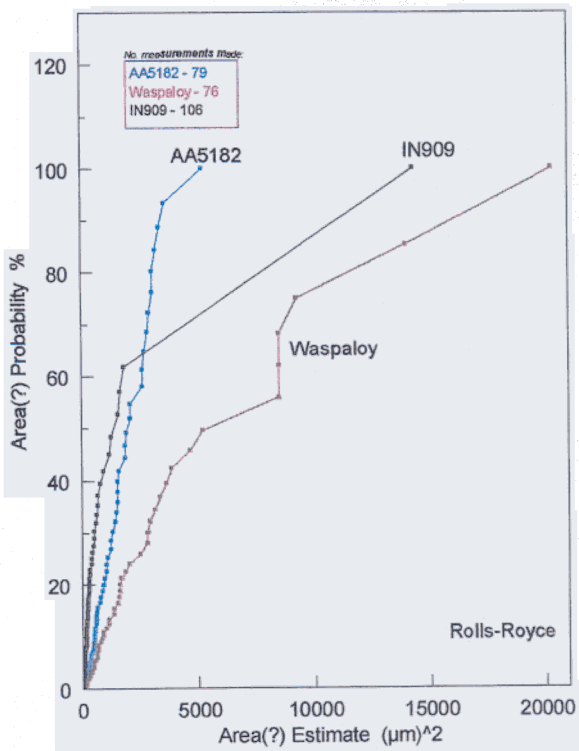
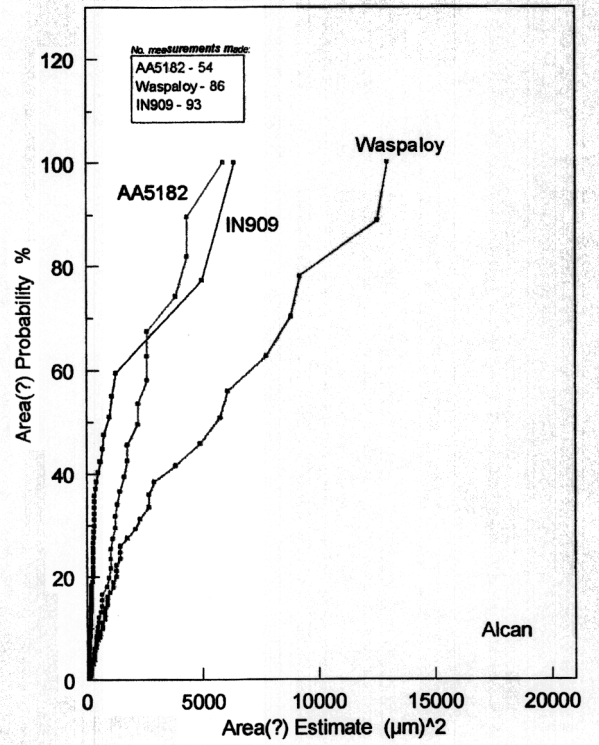
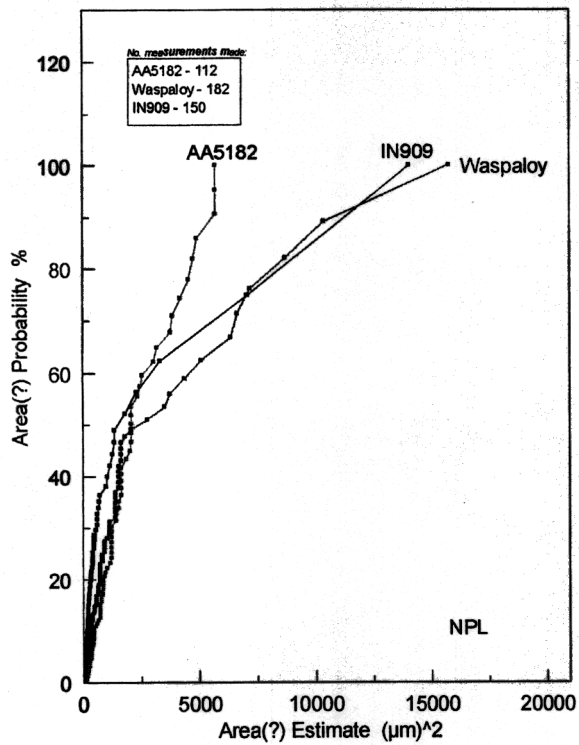
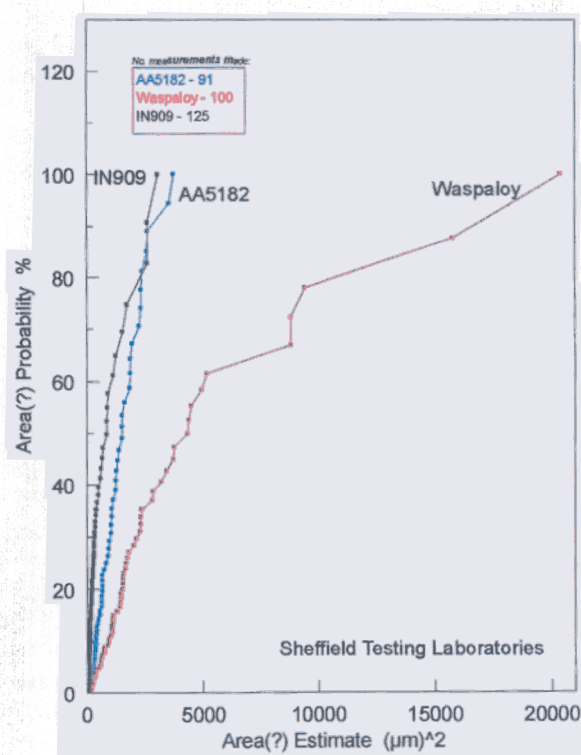
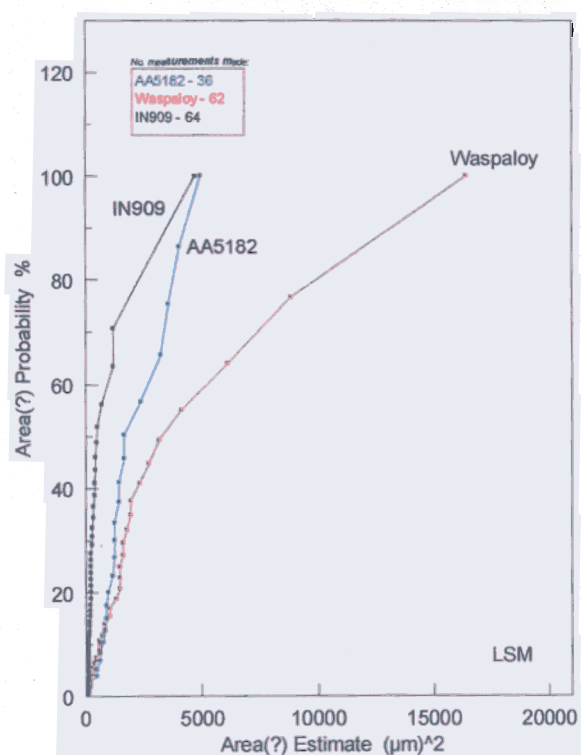
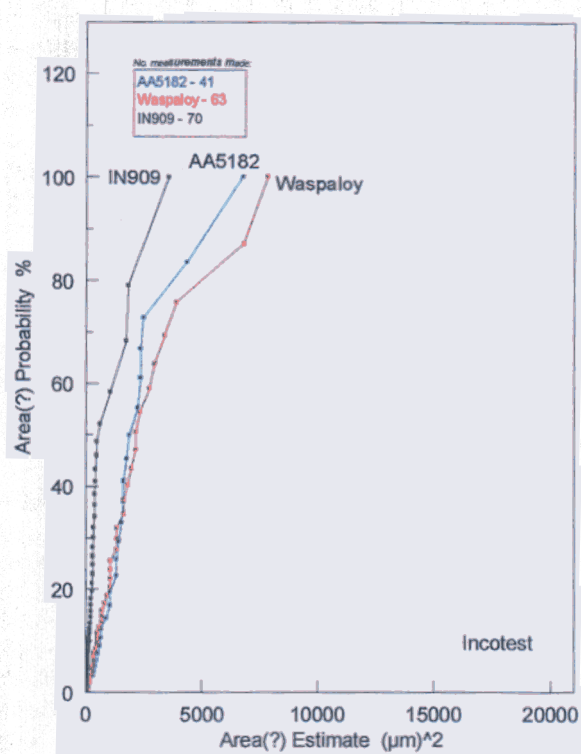
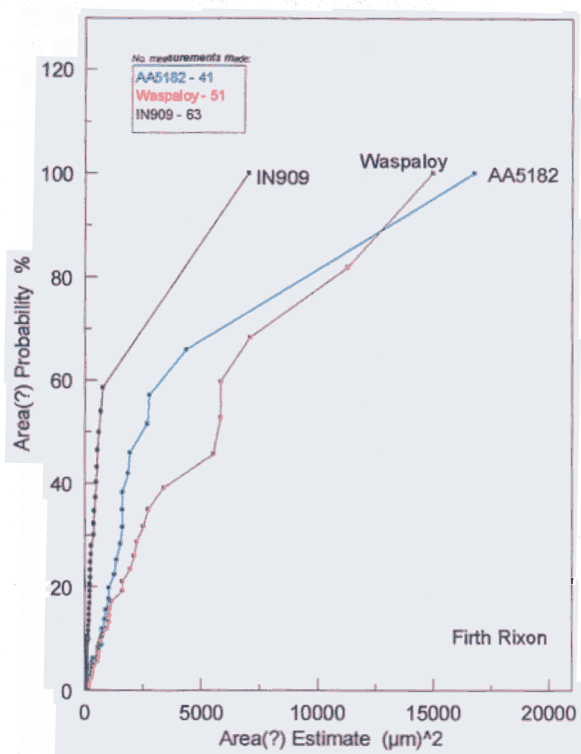
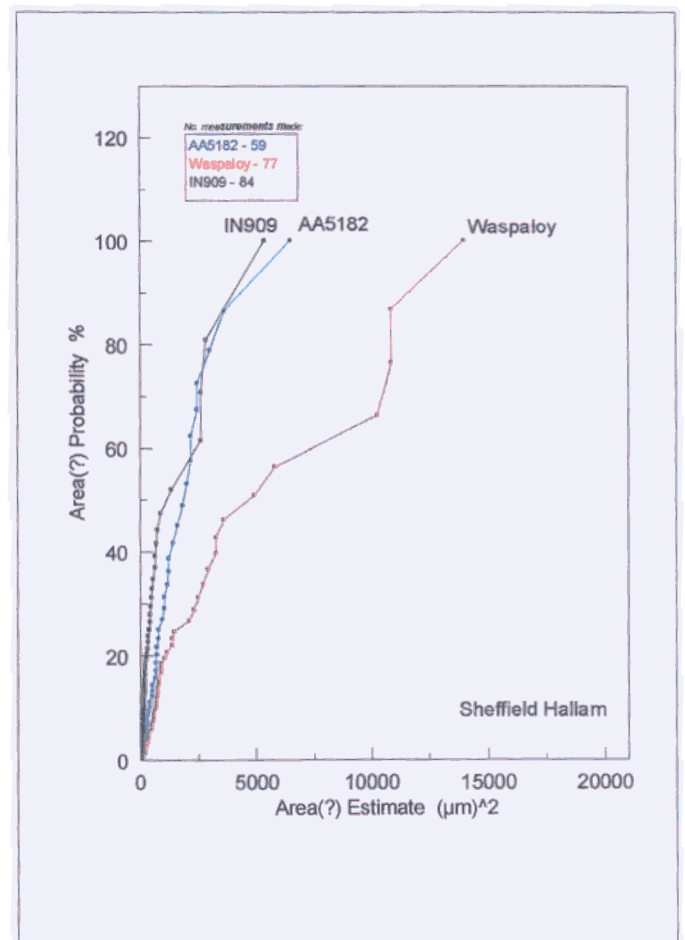
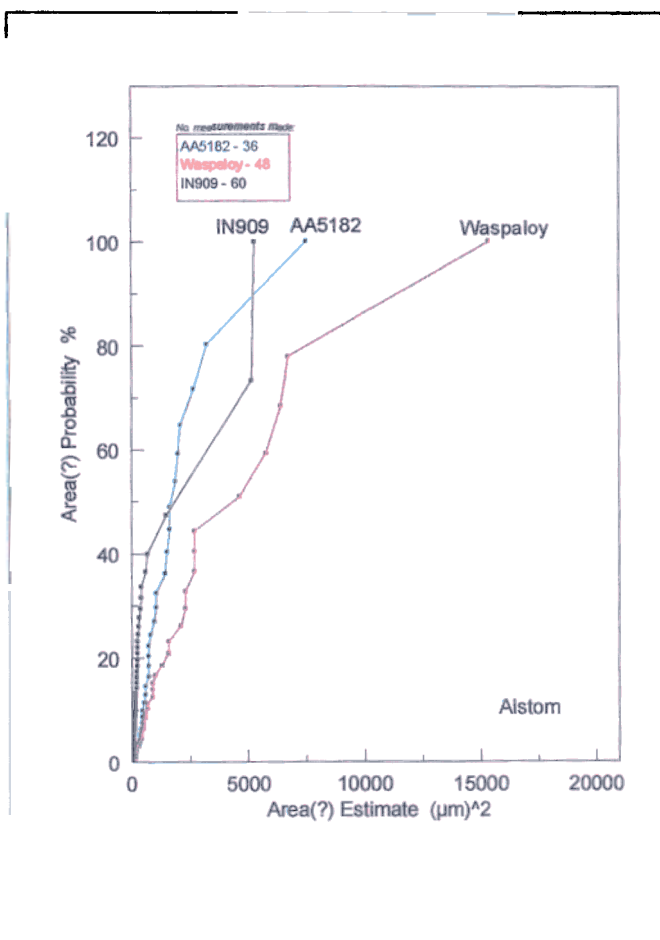
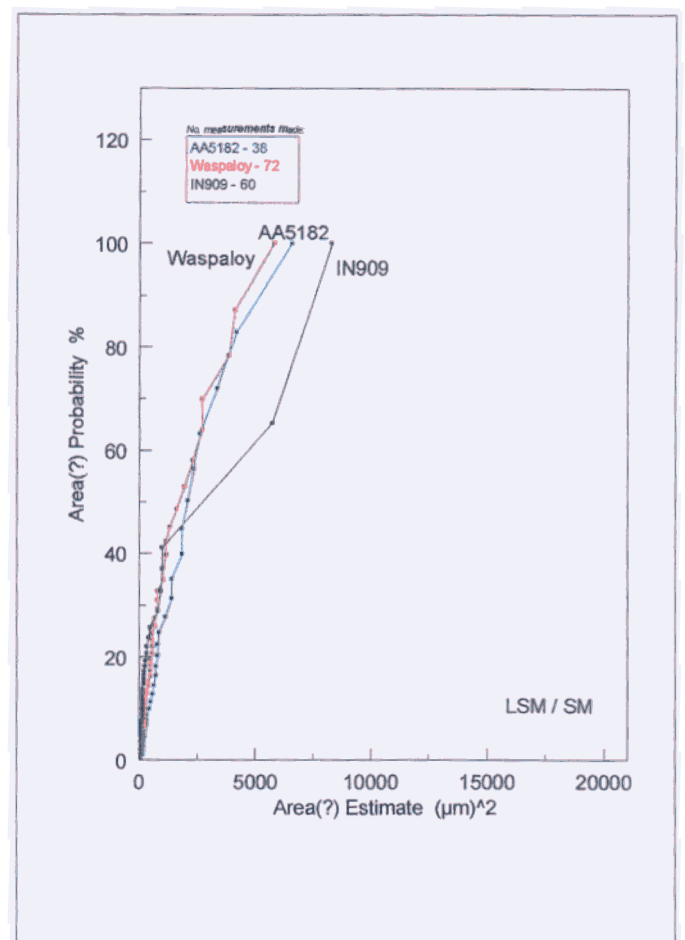
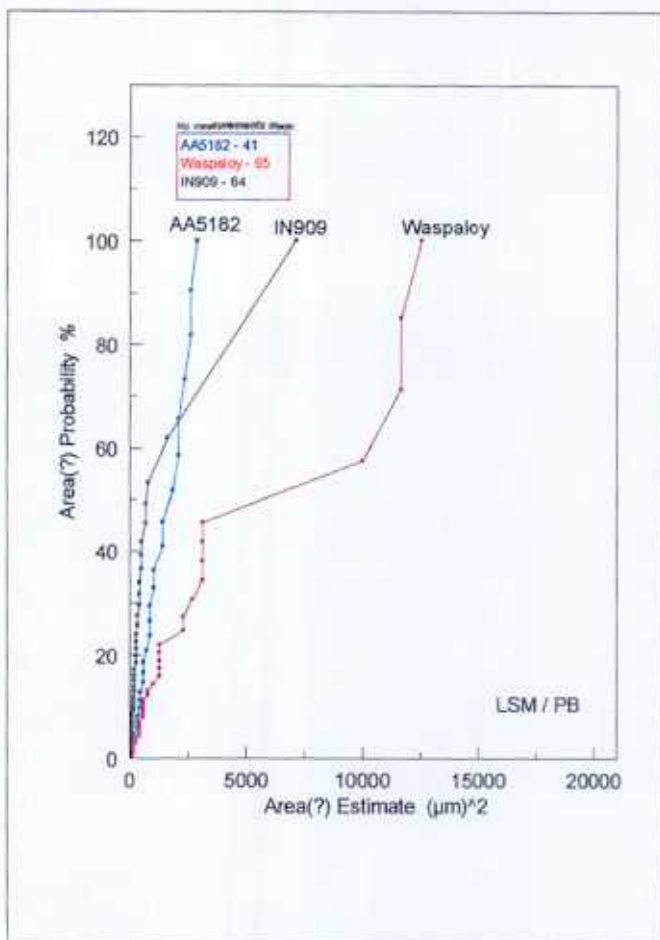


Fig 16 Area (E) probability plotted against Area (E).







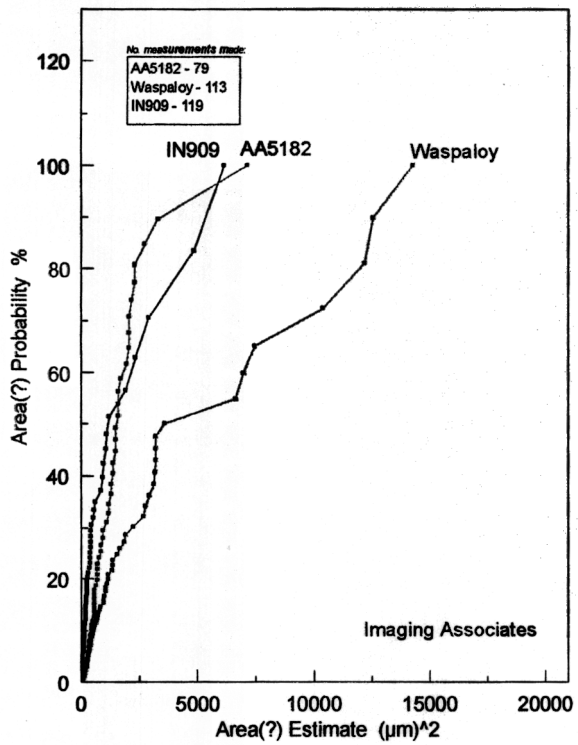
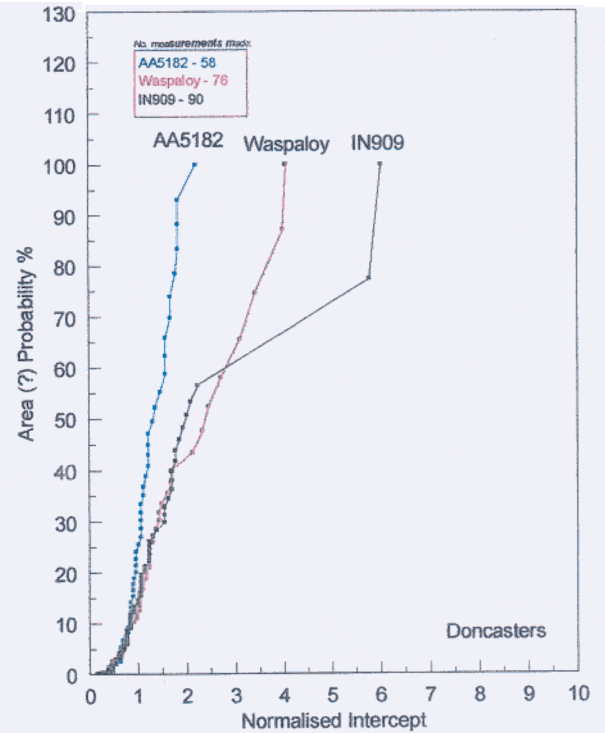
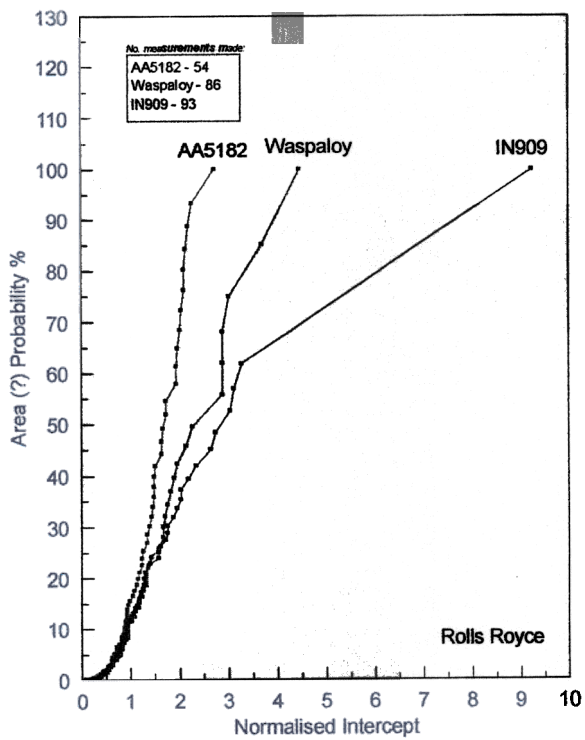
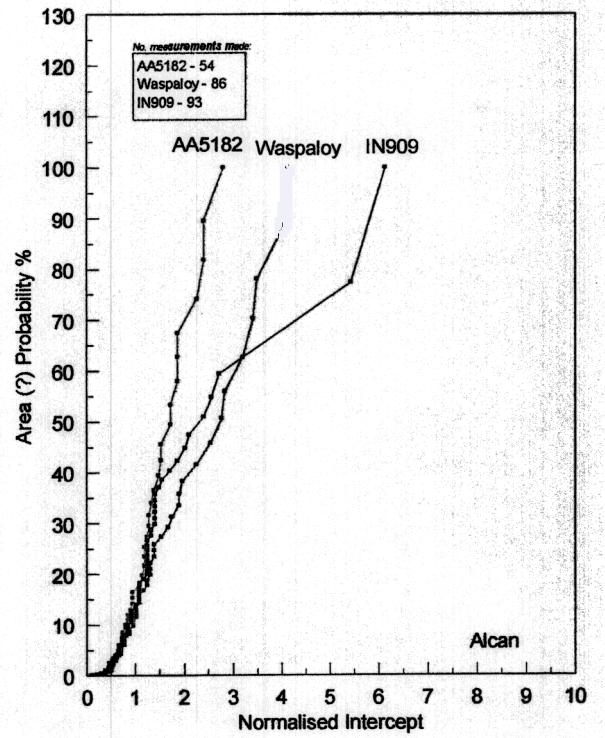
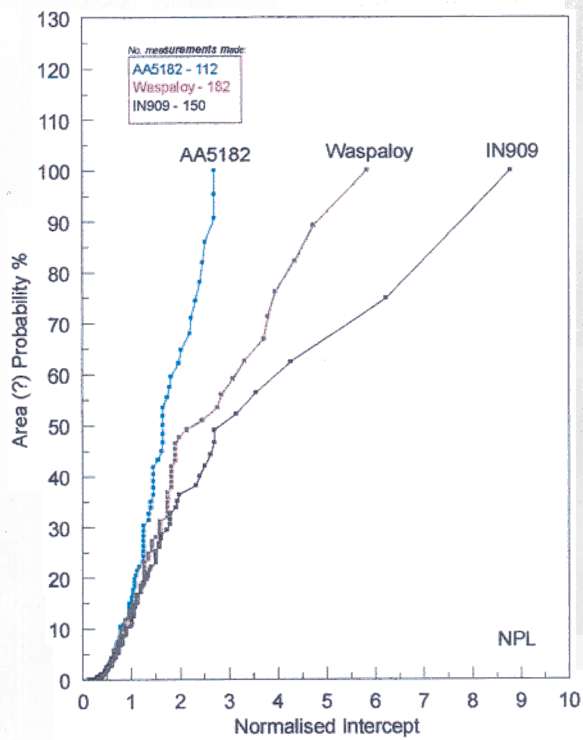
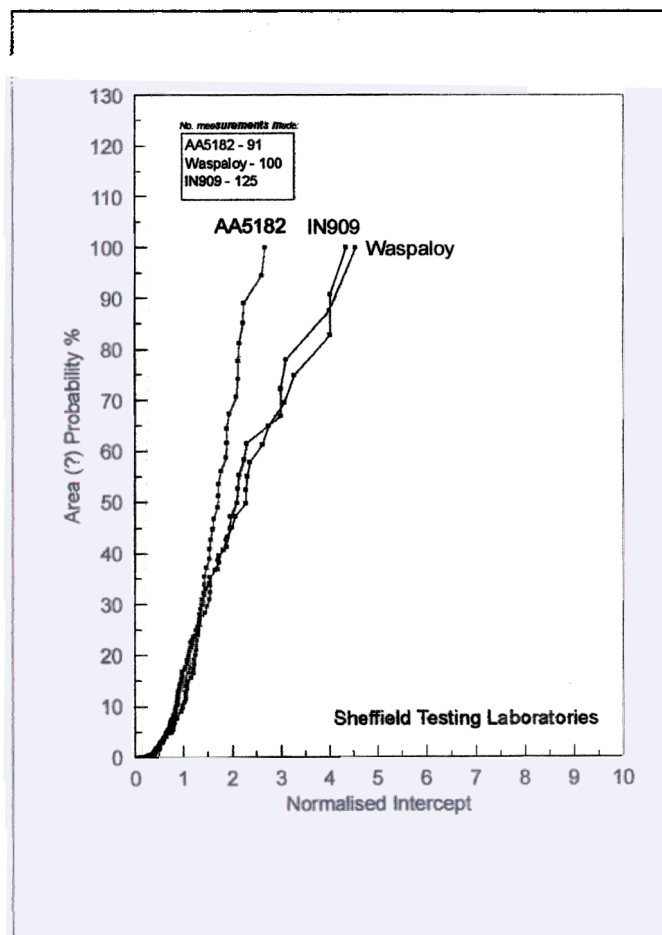
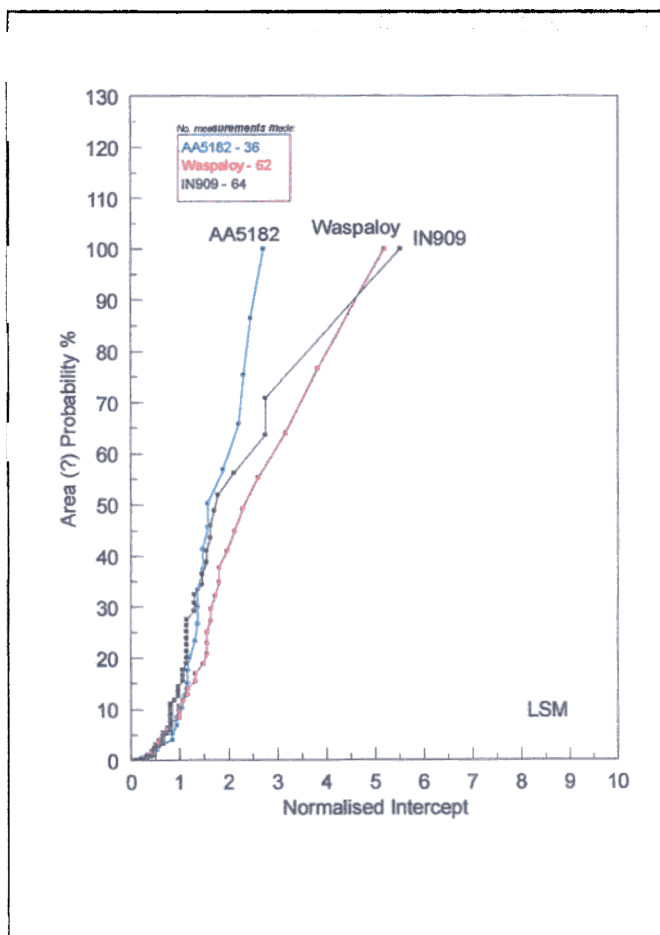
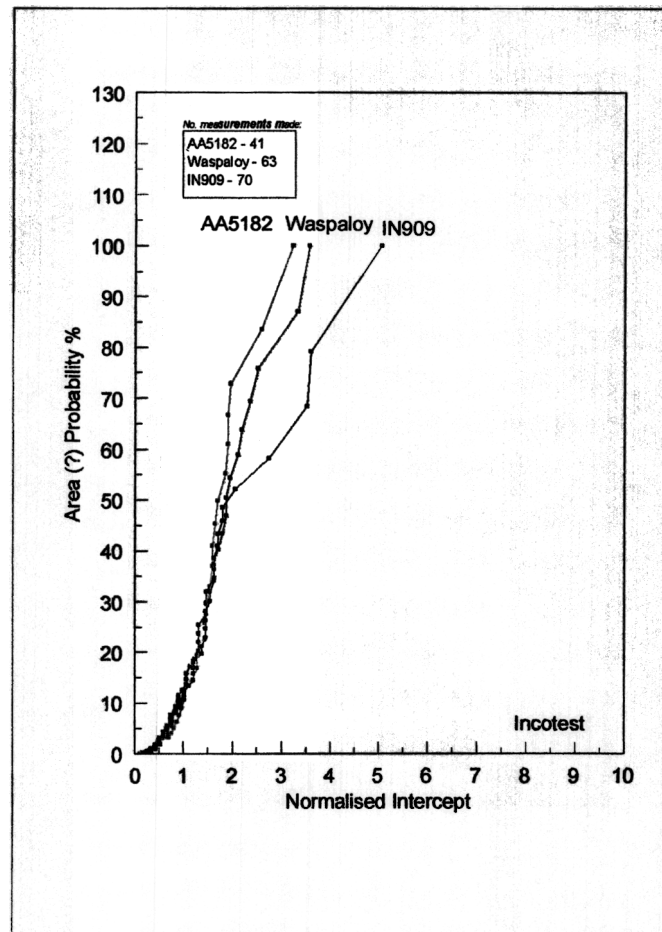
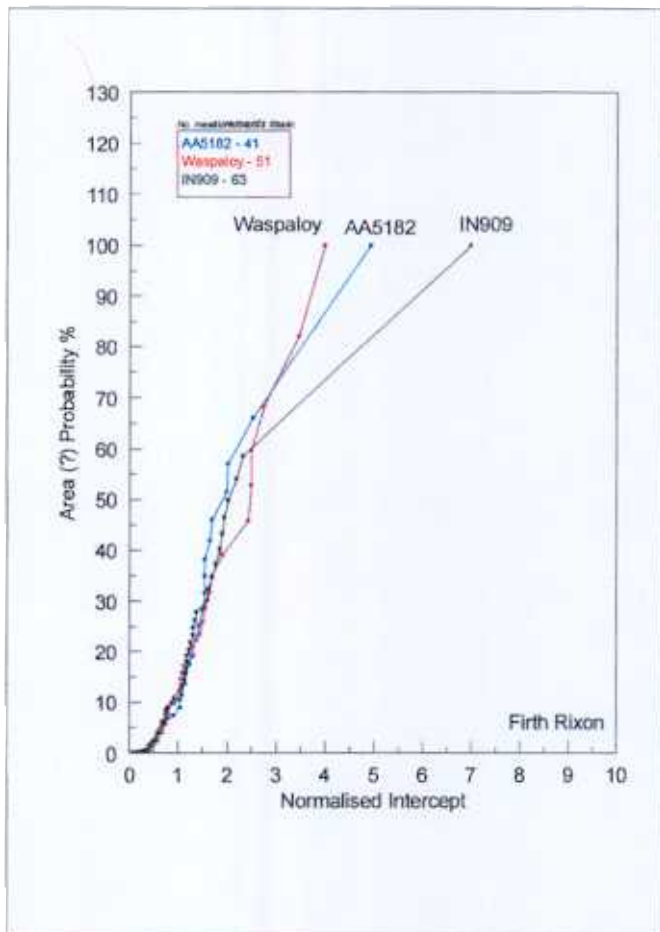
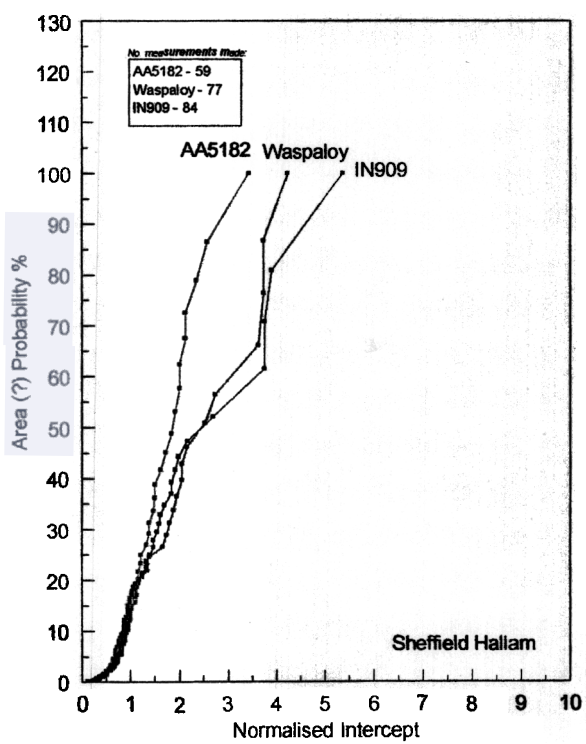
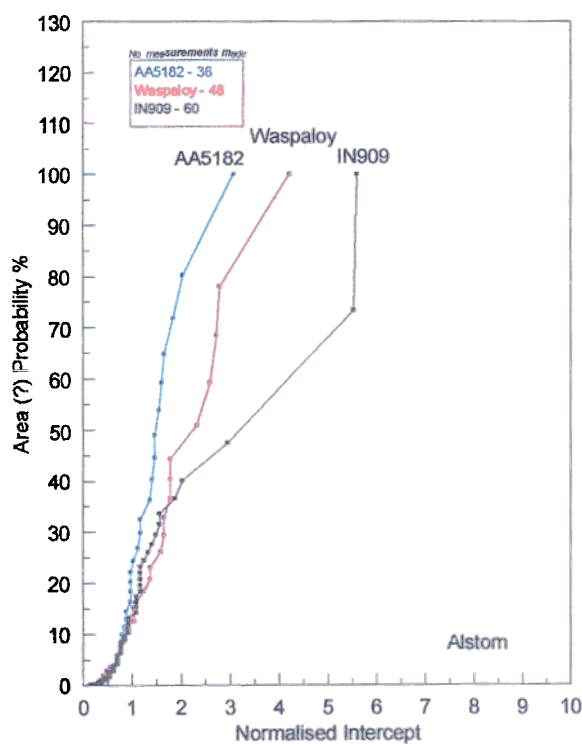
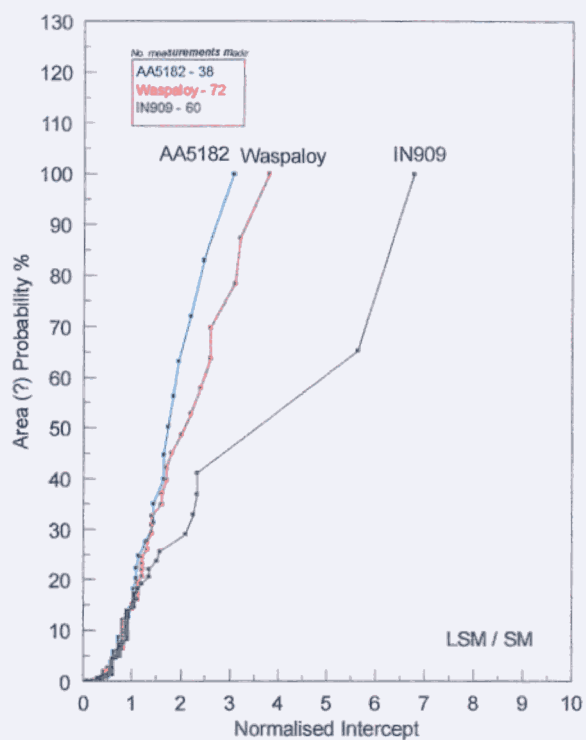
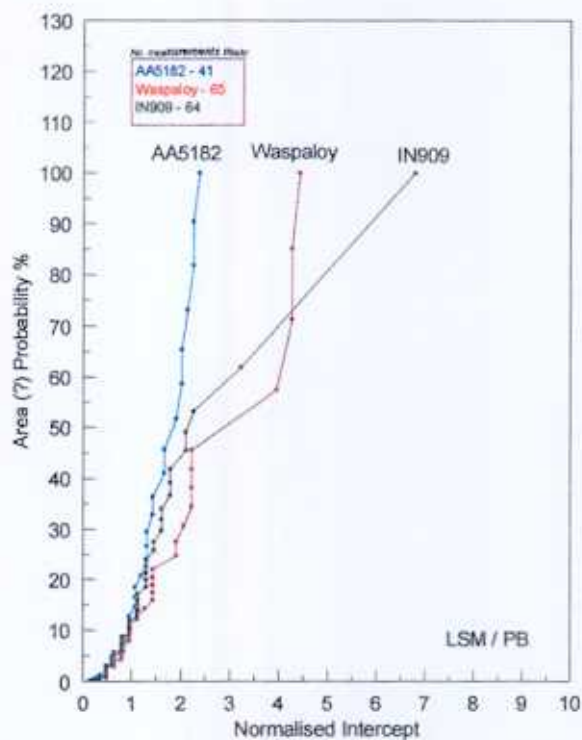
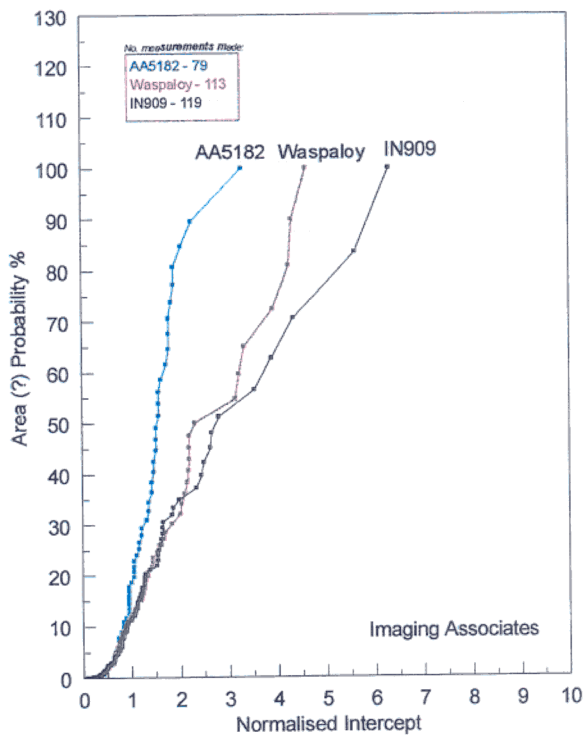


Fig 17 Area (E) probability plotted against Normalised Intercept.









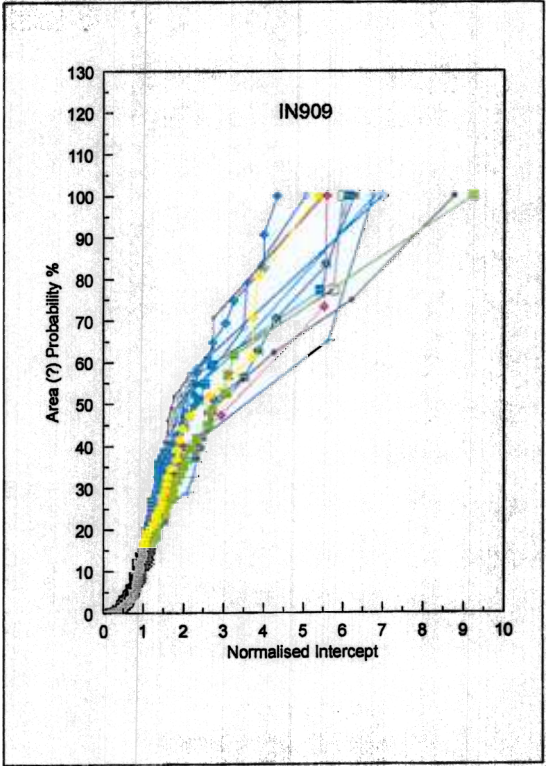
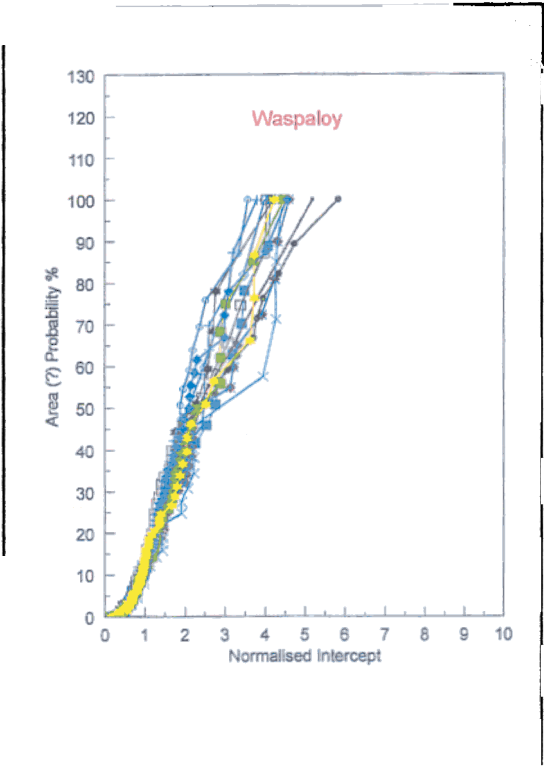
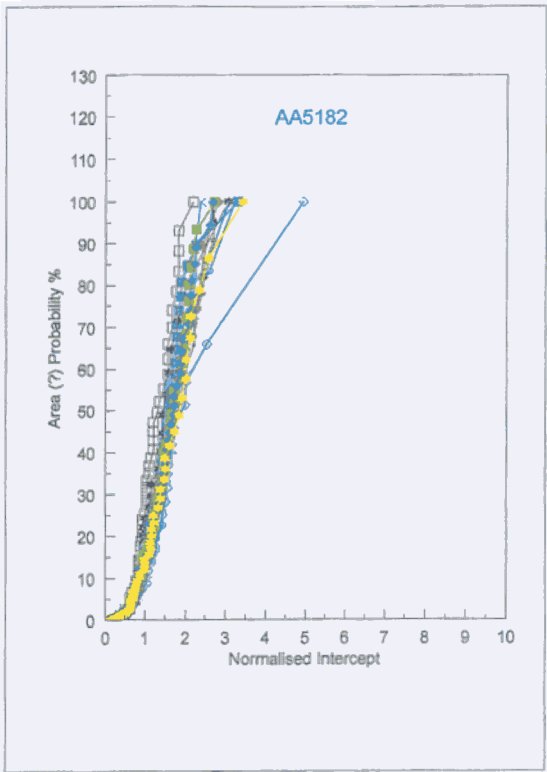
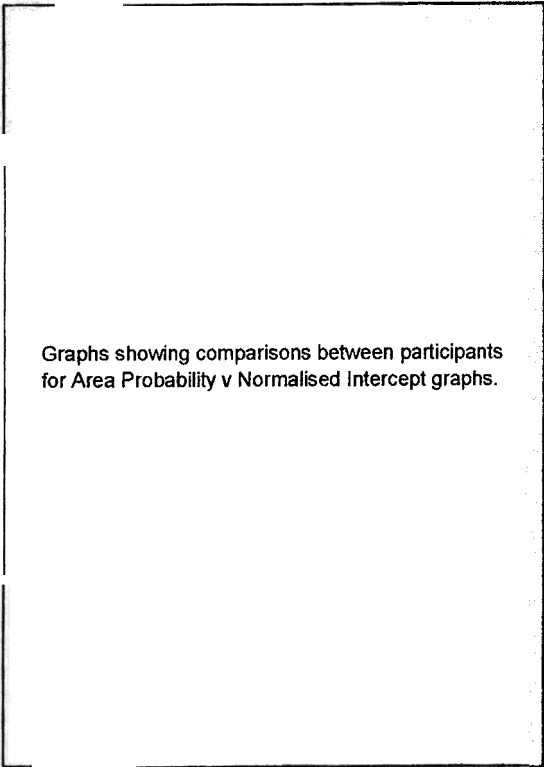
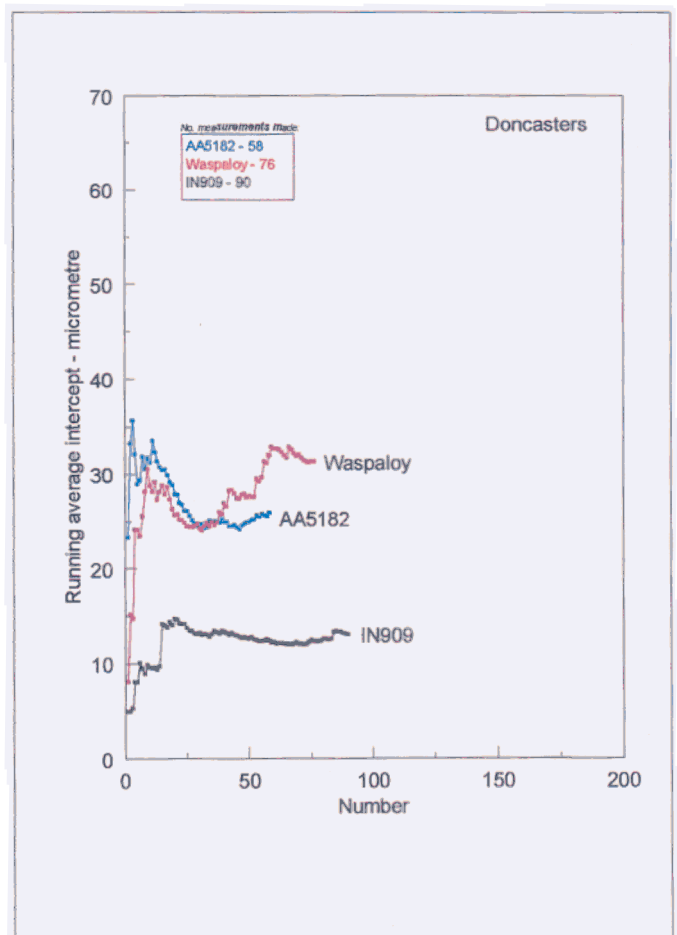
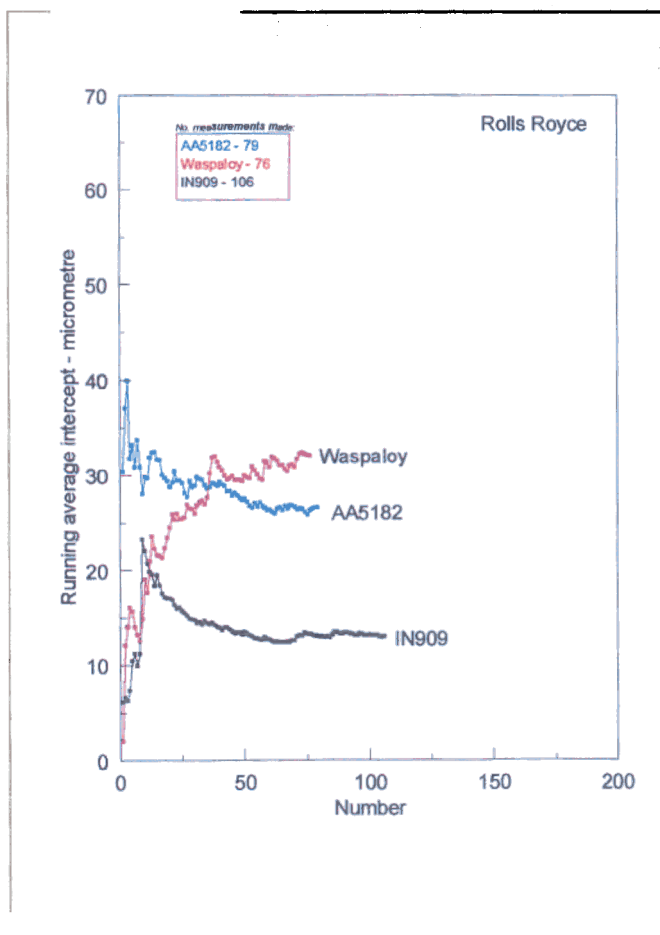
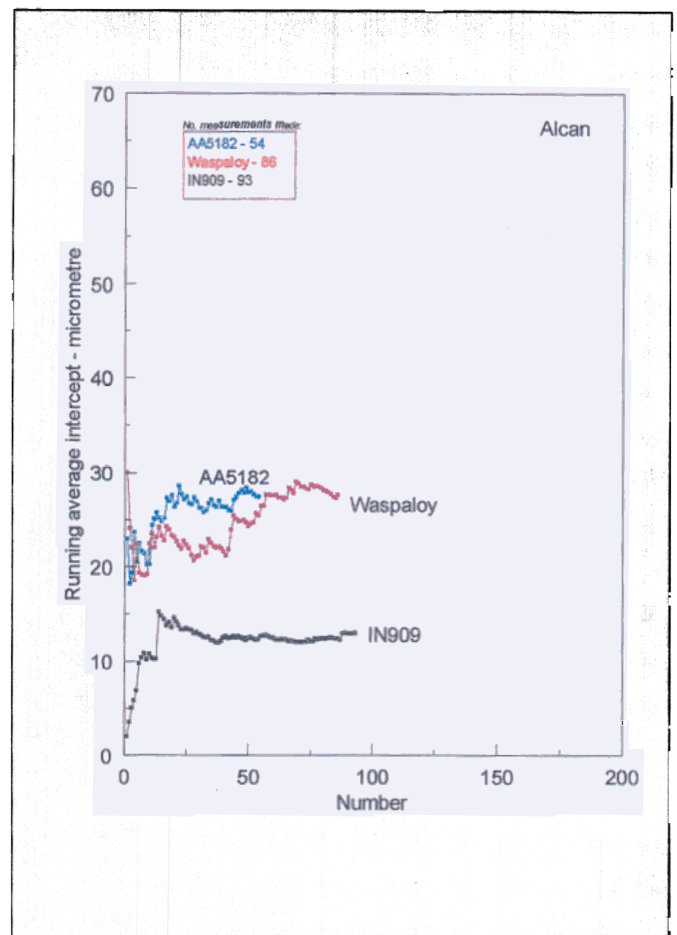
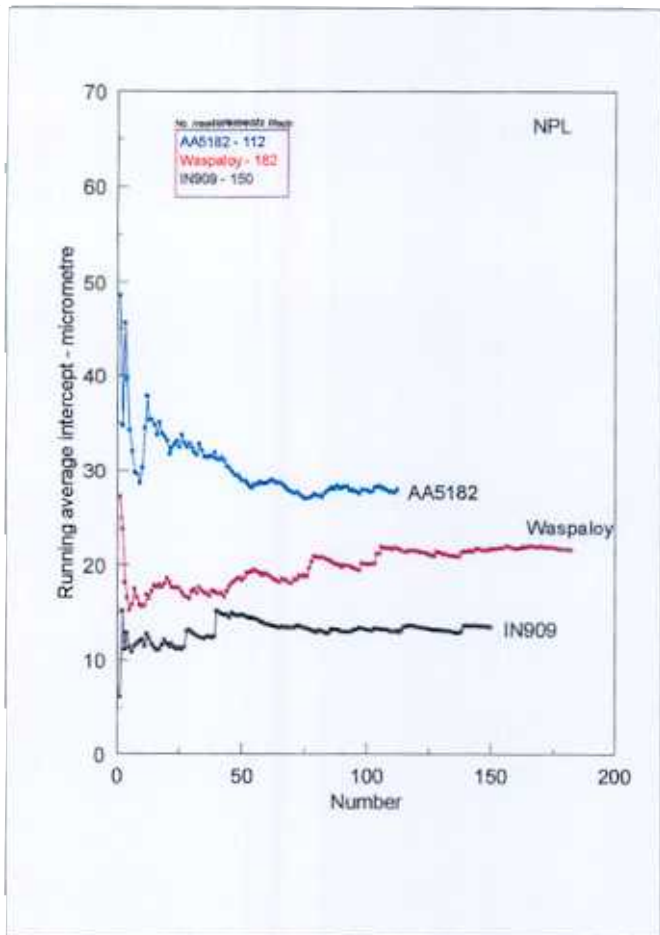
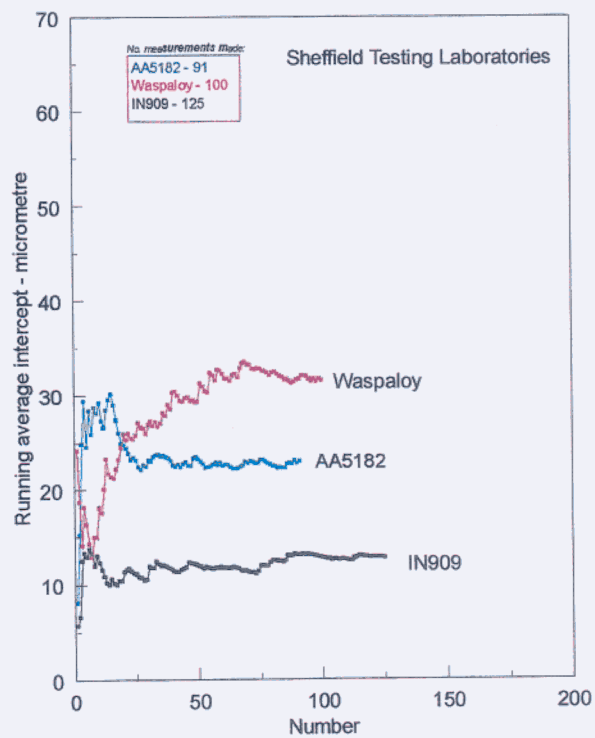
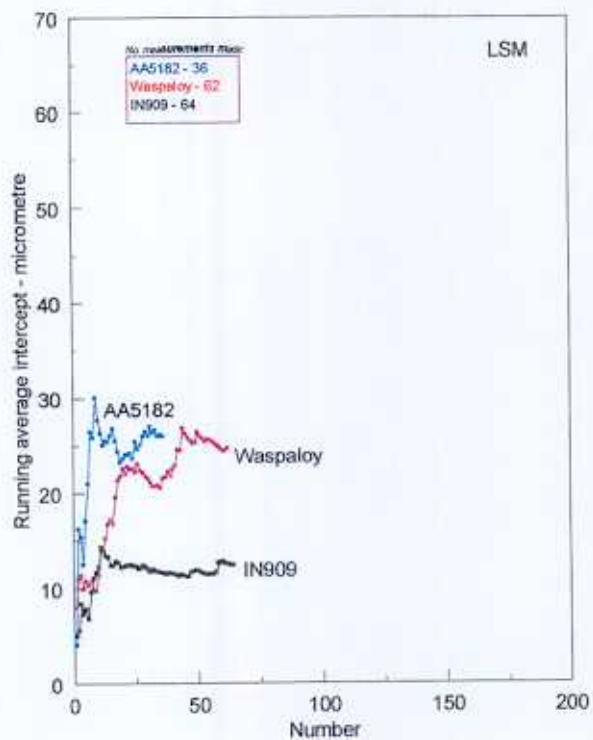
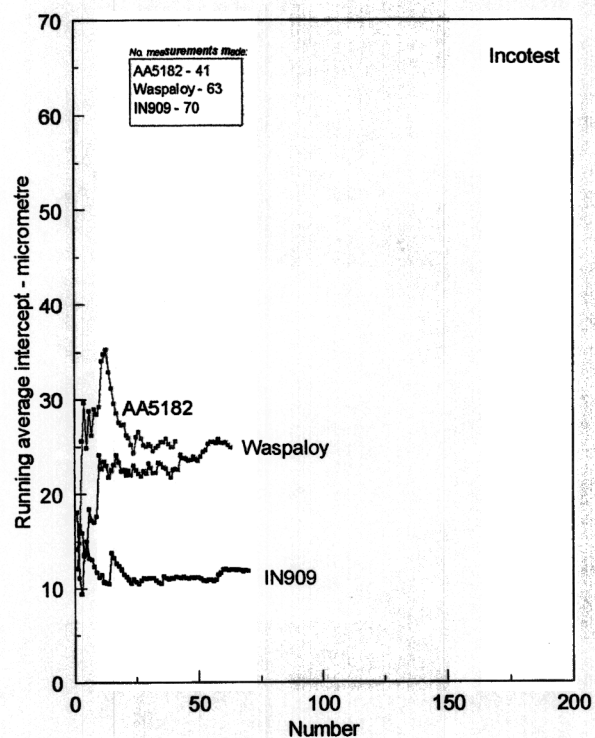
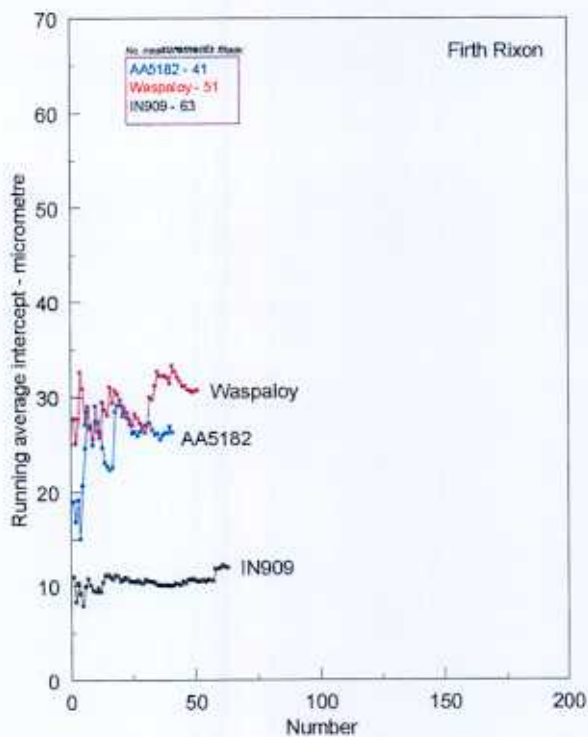
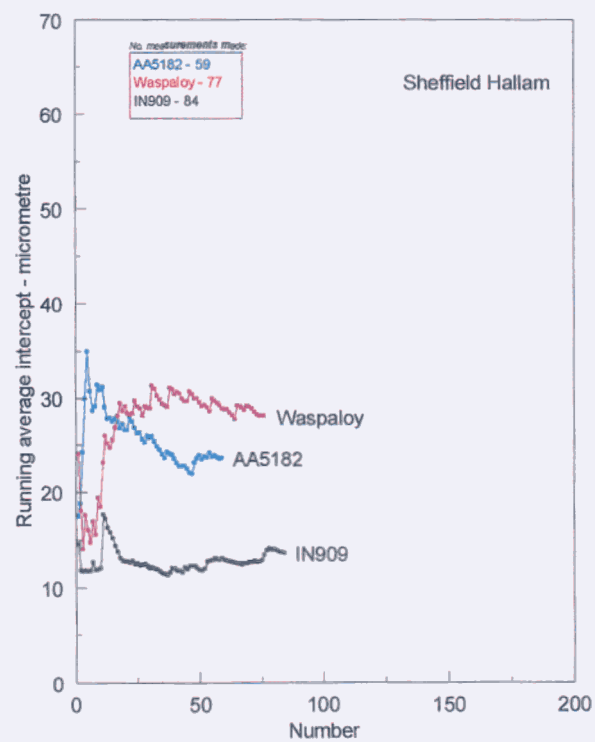
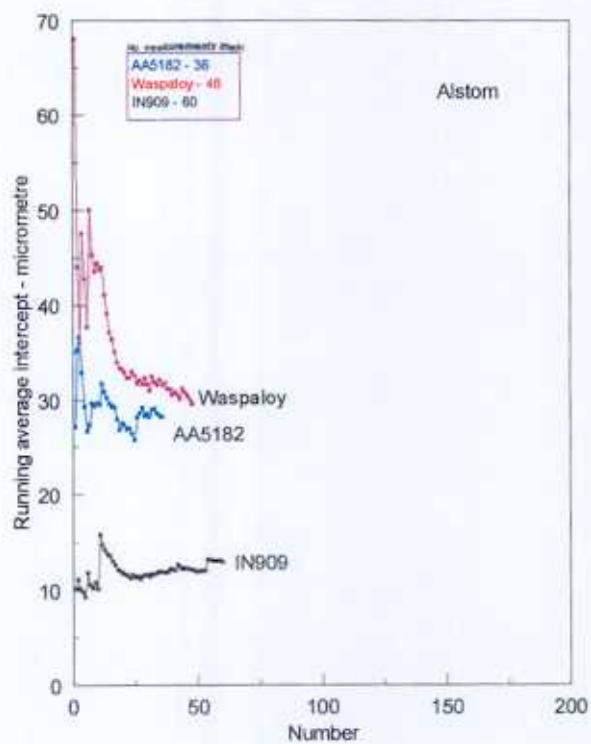
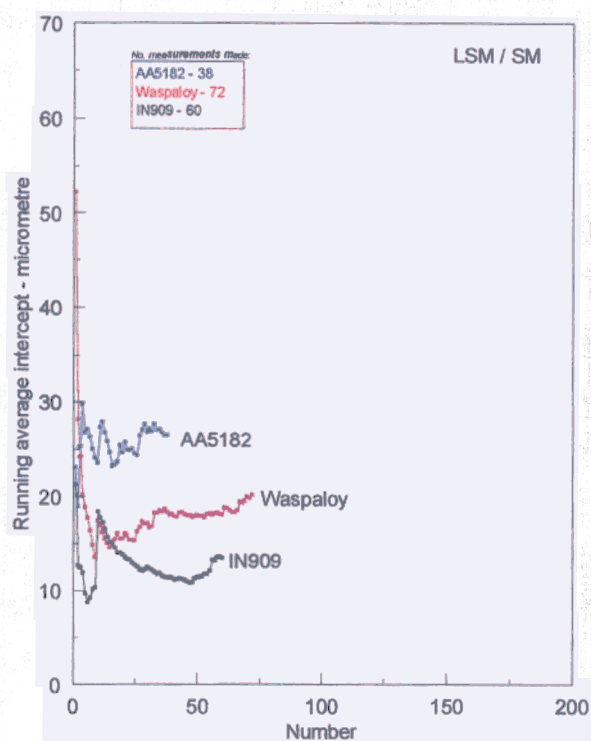
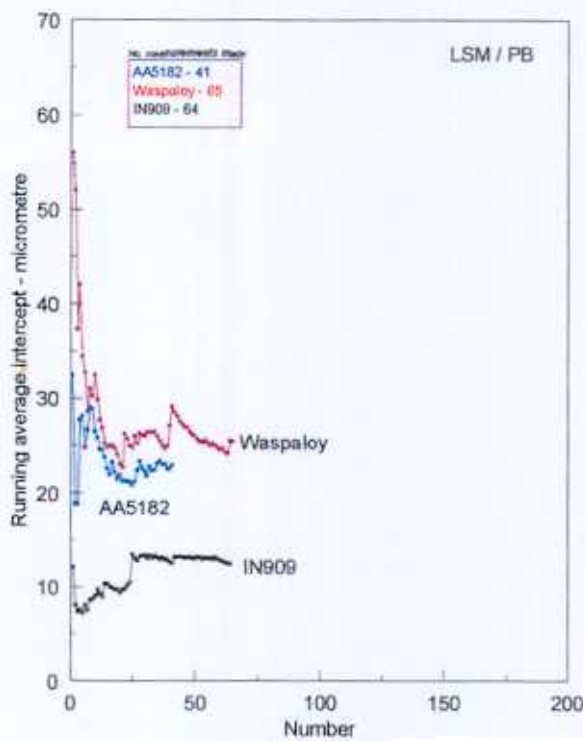


Fig 18 Area (E) probability plotted against Normalised Intercept - all participants for each material.

Fig 19 Running average intercept plotted against Number.







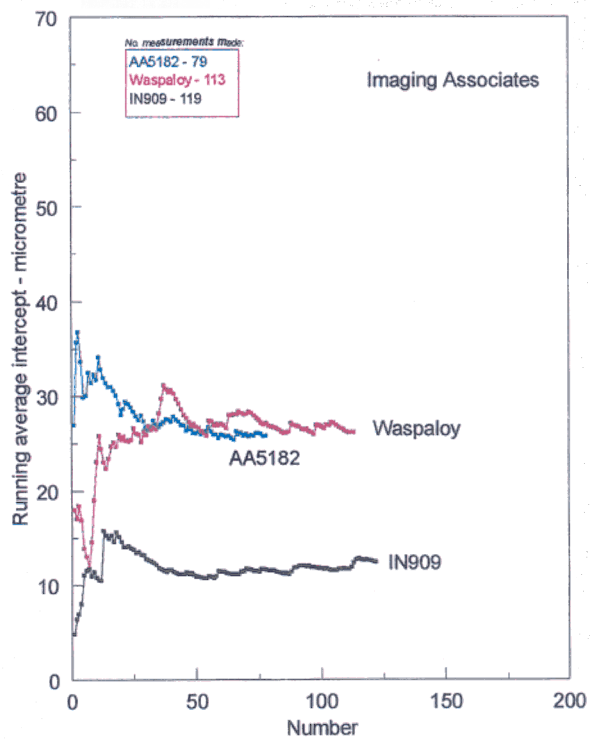
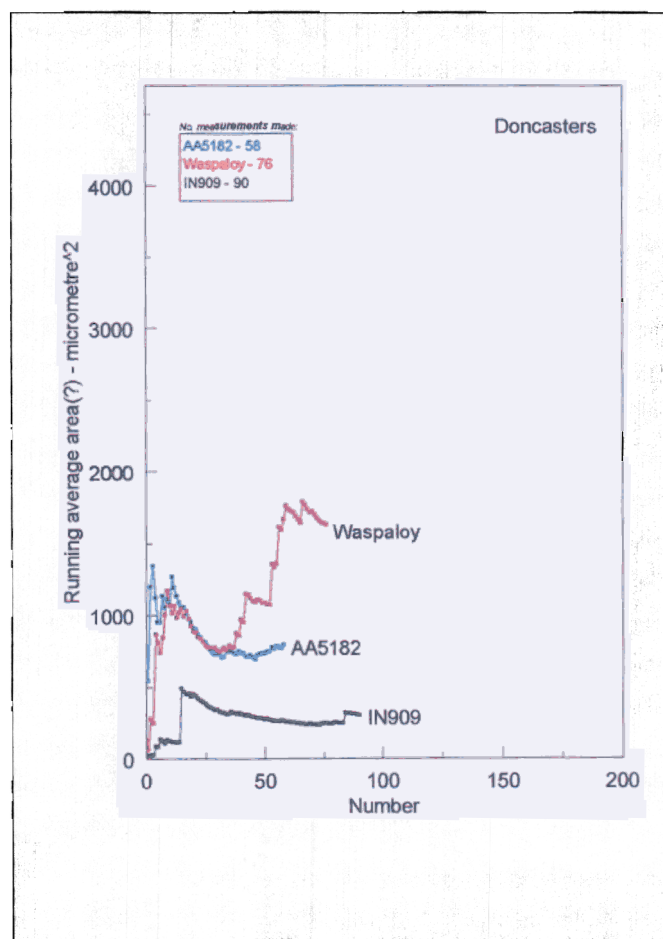
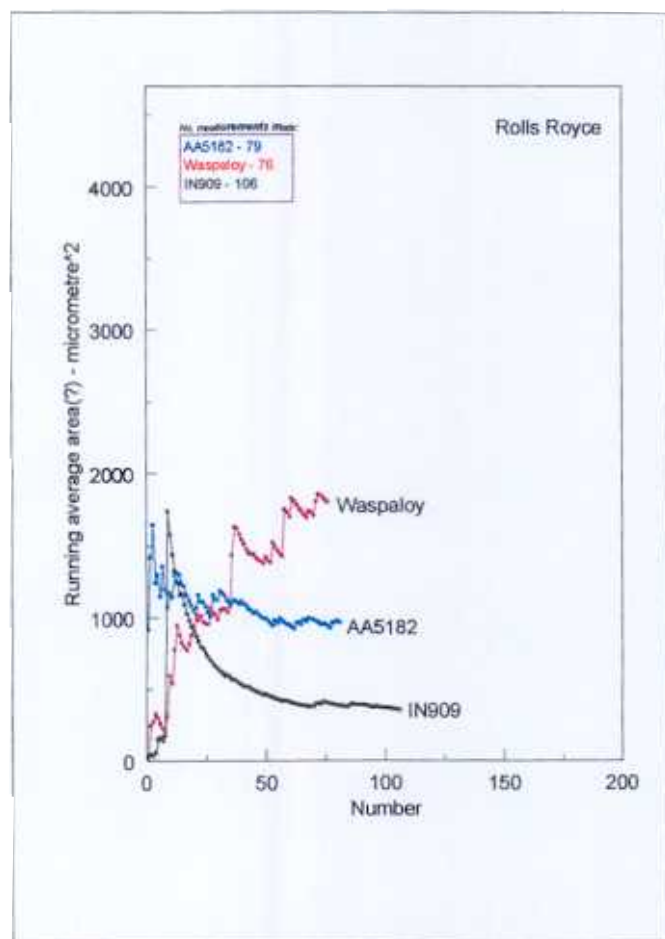
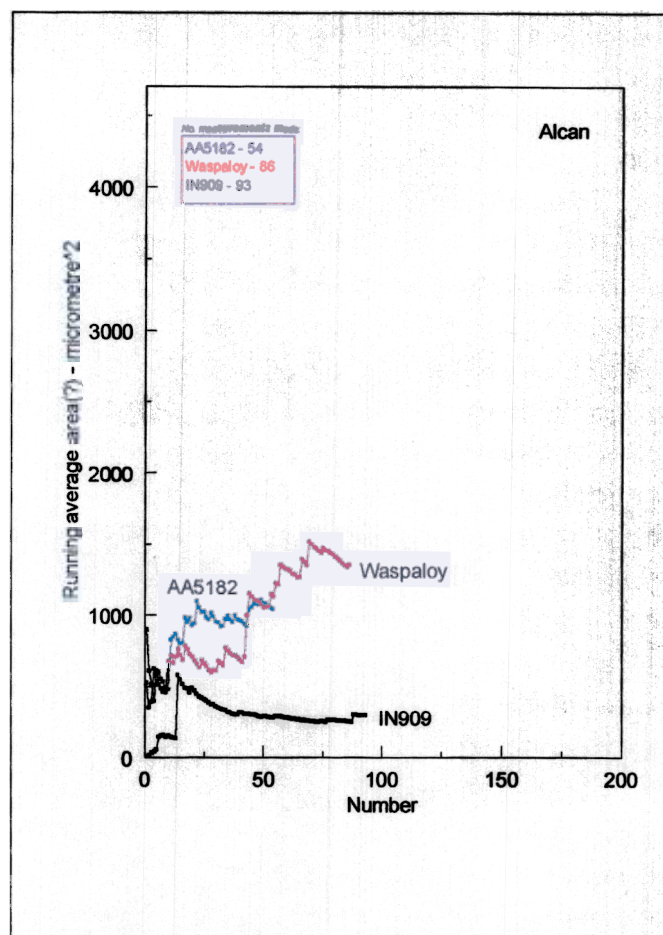
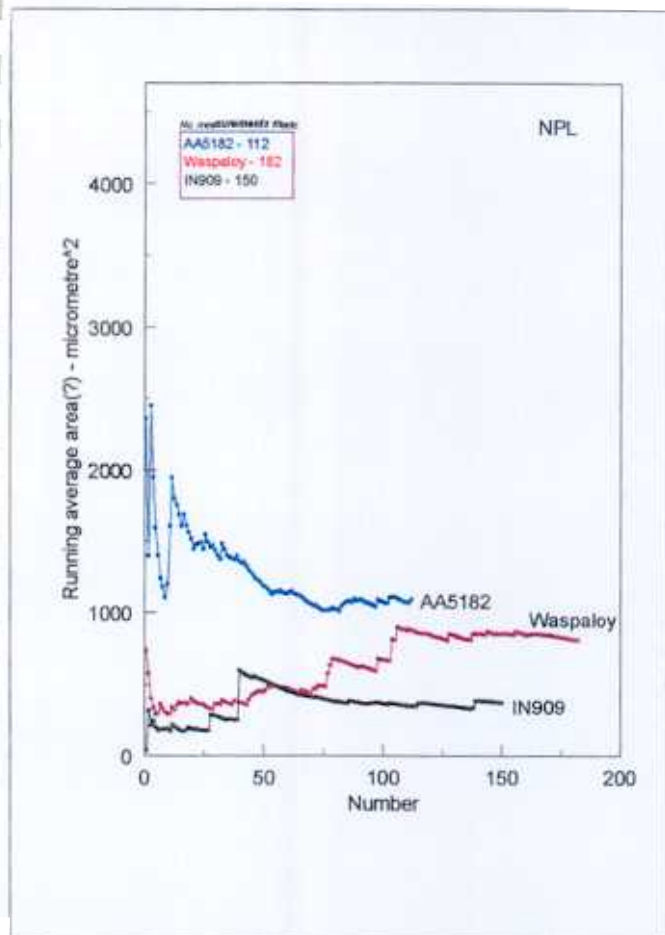
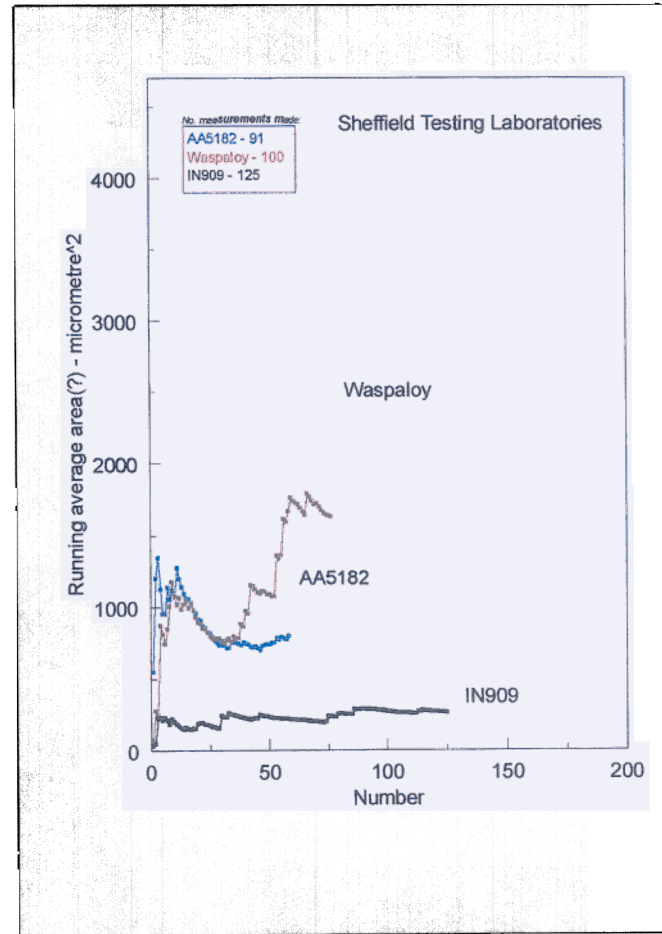
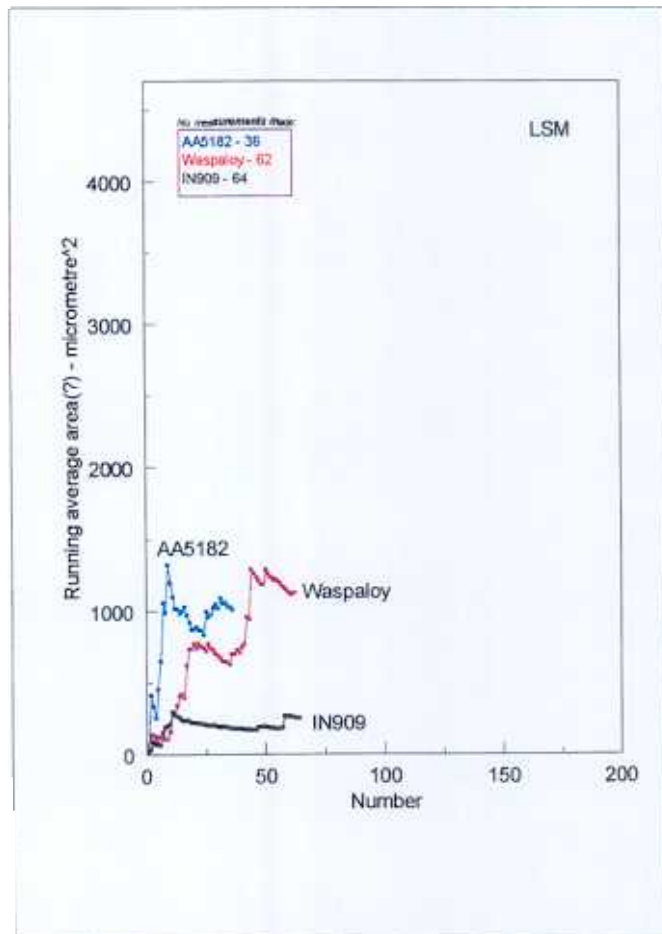
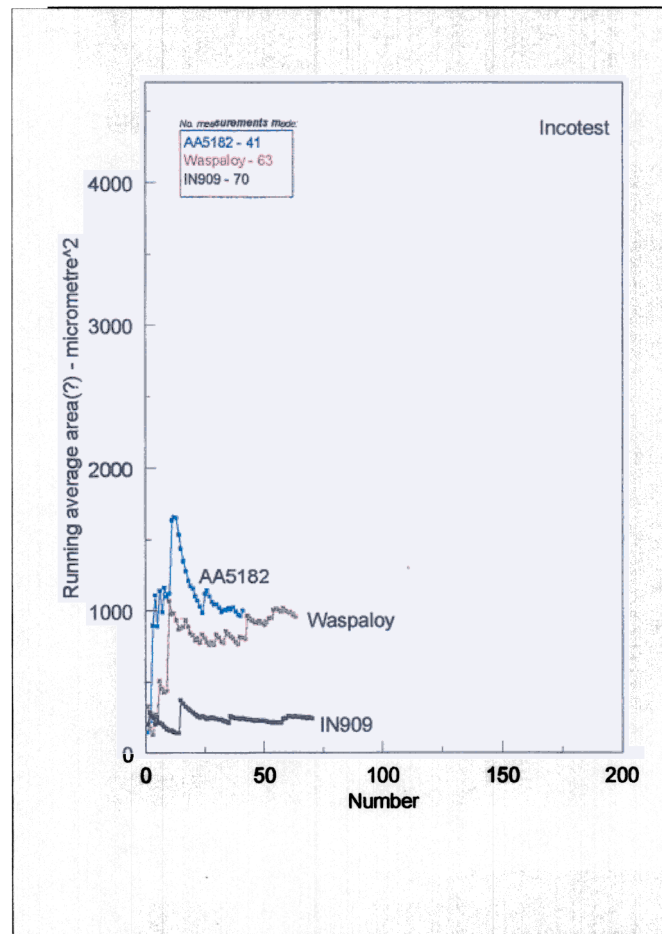
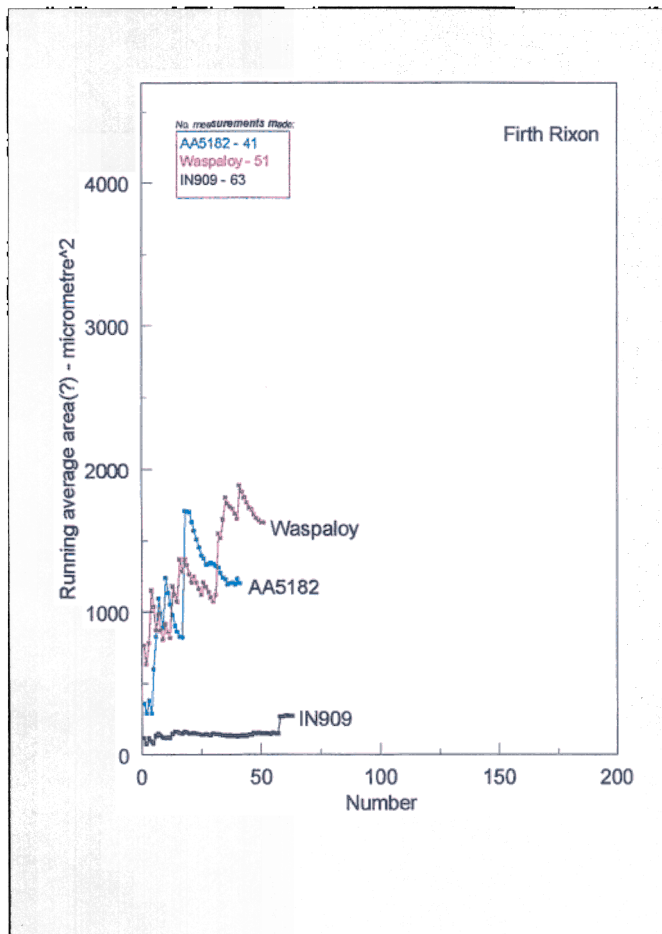
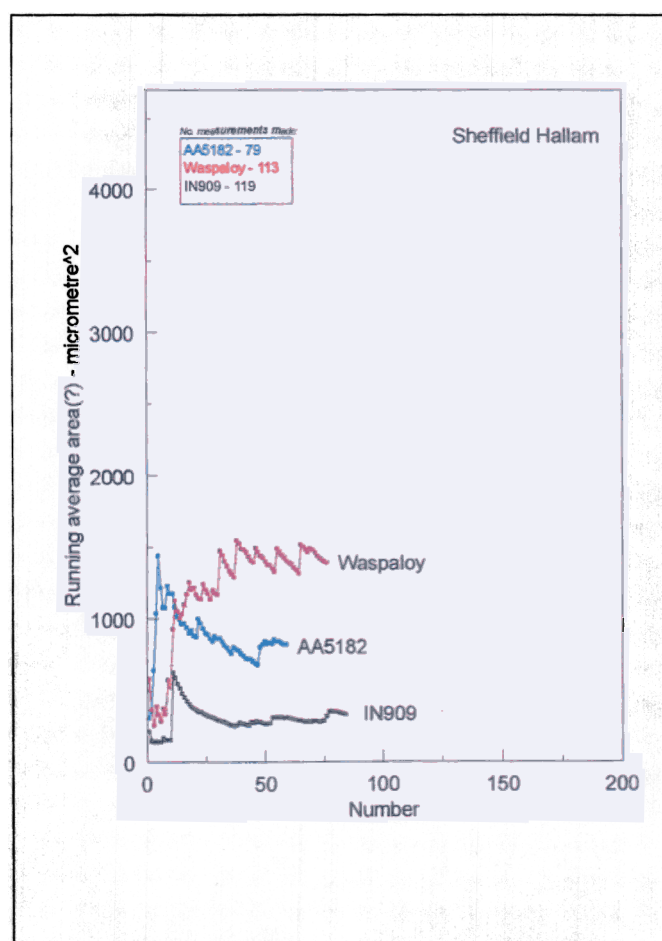
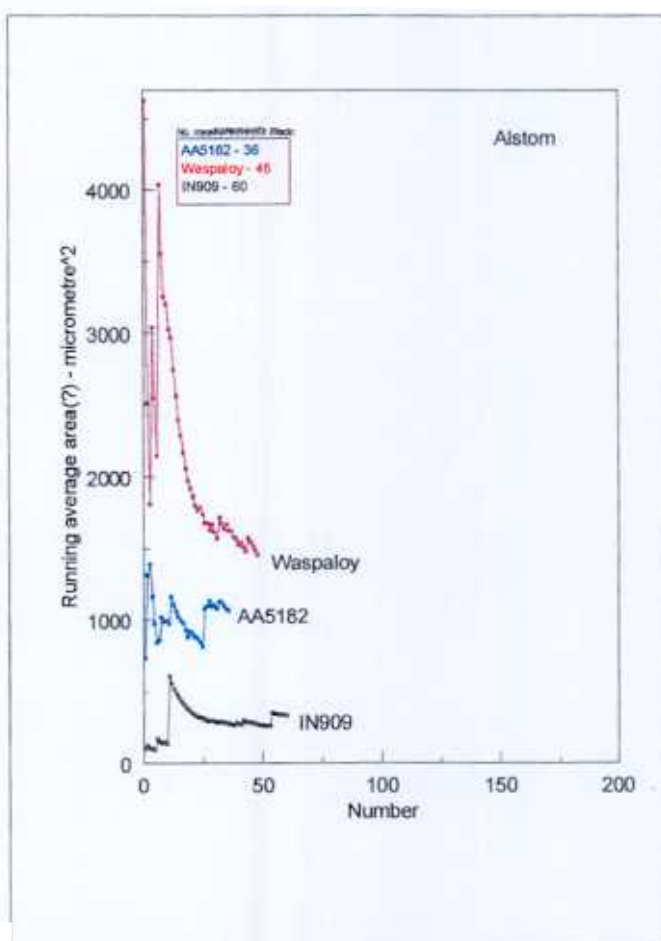
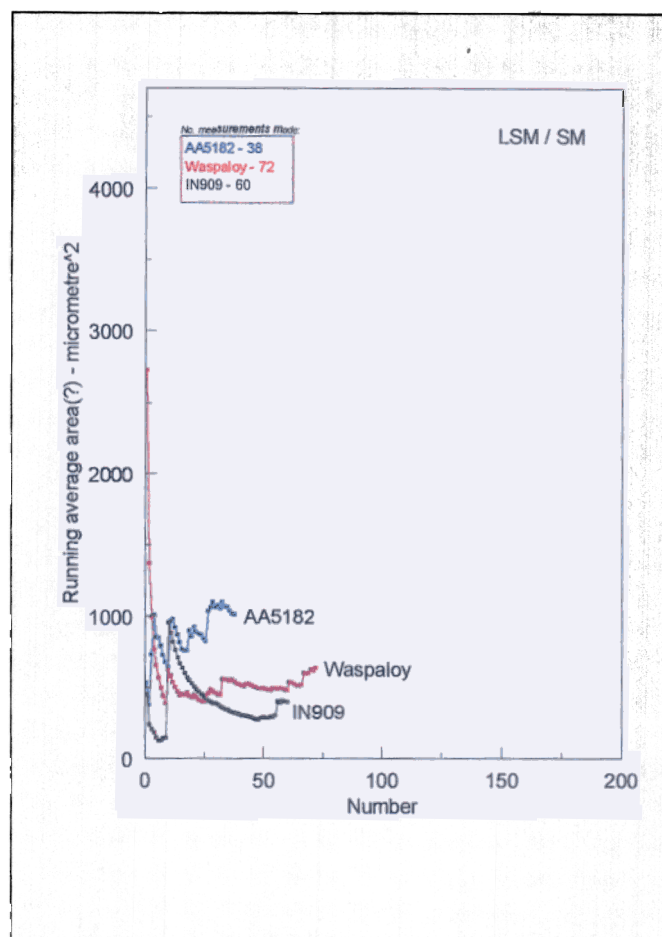
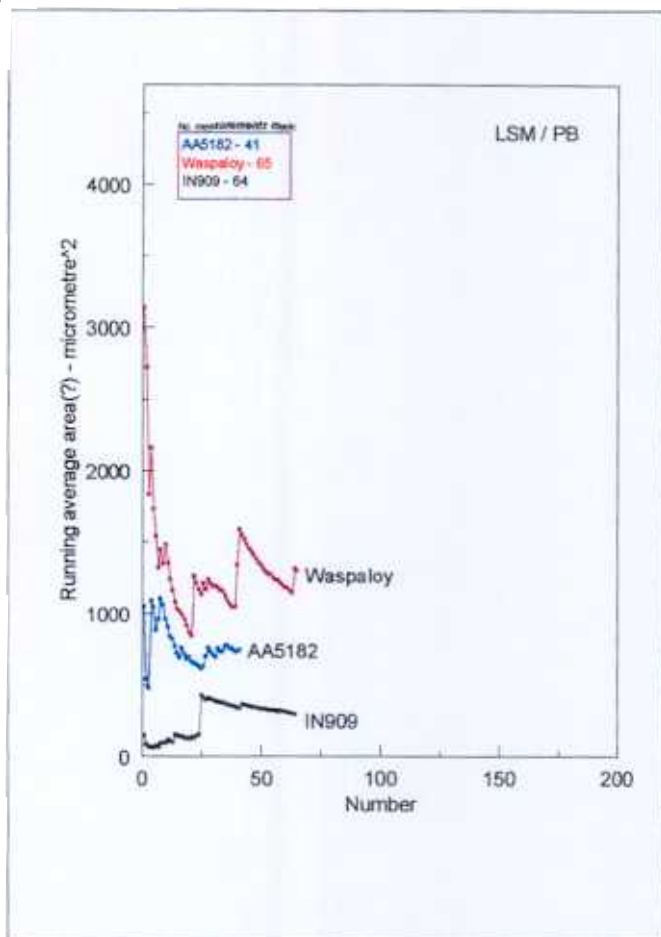
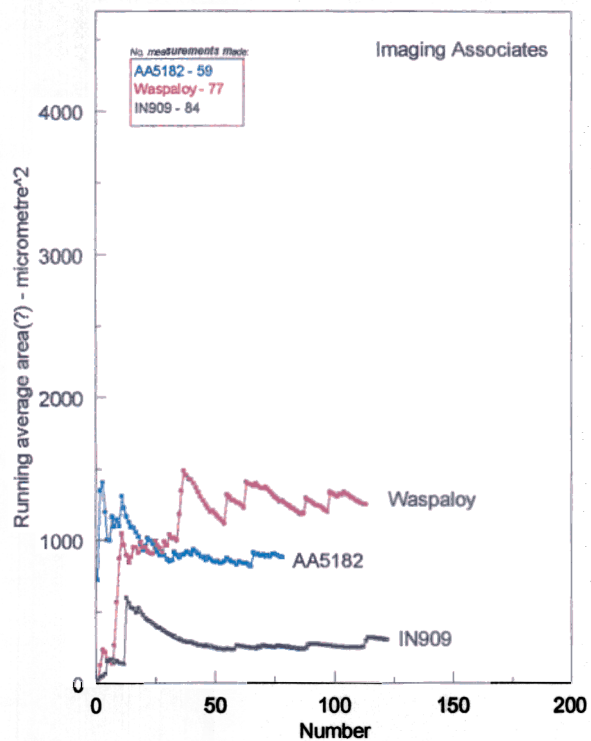


Fig 20 Running average area (E) plotted against Number.

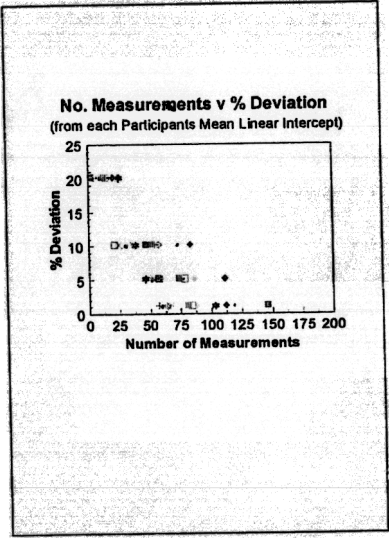




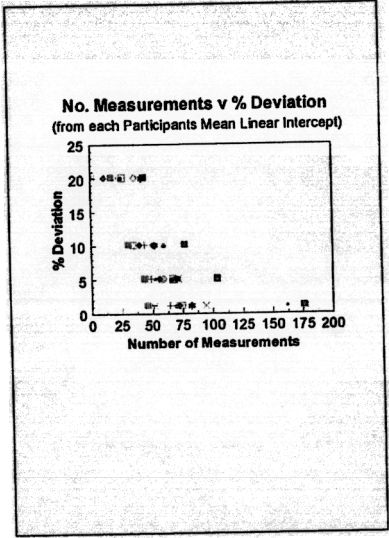




IN909				
Participant	20 %	10%	5%	1%
N.P.L	2	47	57	146
Alcan	14	23	88	88
Rolls Royce	25	38	46	103
Doncasters	14	21	78	84
Firth Ribson	12	57	58	63
Incotest	8	36	57	66
LSM	7	14	57	82
Sheffield Testing Laboratories	17	73	77	119
LSM / PB	22	24	53	62
LSM / SM	12	54	55	57
Alstom	18	31	52	60
Sheffield Hallam University	13	52	74	81
Imaging Associates	20	83	111	112



Waspeloy				
Participant	20 %	10%	5%	1%
N.P.L	44	77	104	176
Alcan	42	52	70	82
Rolls Royce	26	35	67	74
Doncasters	38	51	59	70
Firth Ribson	1	31	44	51
Incotest	9	40	50	62
LSM	17	48	53	162
Sheffield Testing Laboratories	21	37	71	84
LSM / PB	11	44	49	64
LSM / SM	25	60	66	70
Alstom	17	31	44	48
Sheffield Hallam University	11	40	56	72
Imaging Associates	10	43	76	105



AA5182				
Participant	20 %	10%	5%	1%
N.P.L	28	44	48	112
Alcan	11	12	43	52
Rolls Royce	17	43	48	76
Doncasters	14	28	46	58
Firth Ribson	5	21	25	41
Incotest	16	19	21	41
LSM	6	19	27	34
Sheffield Testing Laboratories	16	20	21	87
LSM / PB	10	12	26	40
LSM / SM	2	17	26	35
Alstom	5	14	25	35
Sheffield Hallam University	13	31	47	50
Imaging Associates	16	22	43	76

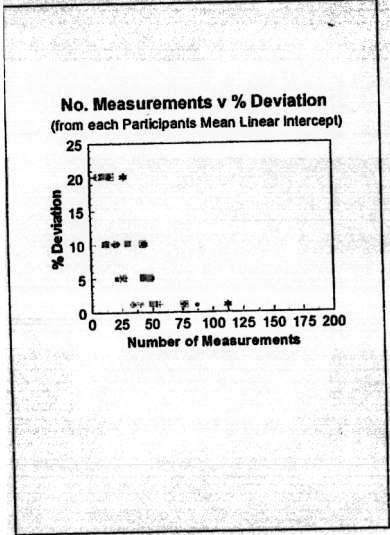


Fig 21 Percentage deviation from running average plotted against Number for each participant.

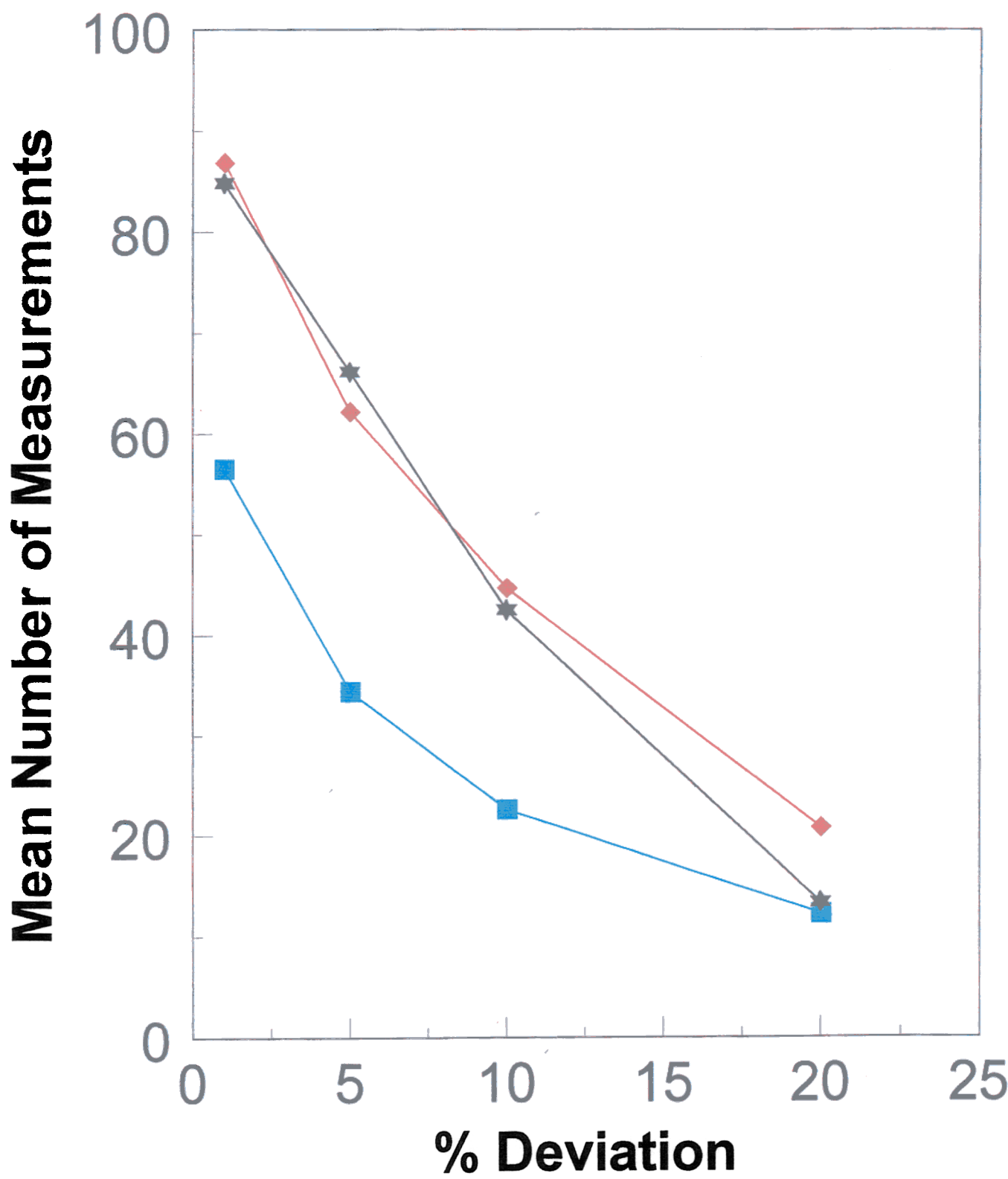


Fig 22 Mean percentage deviation from running average plotted against Number.

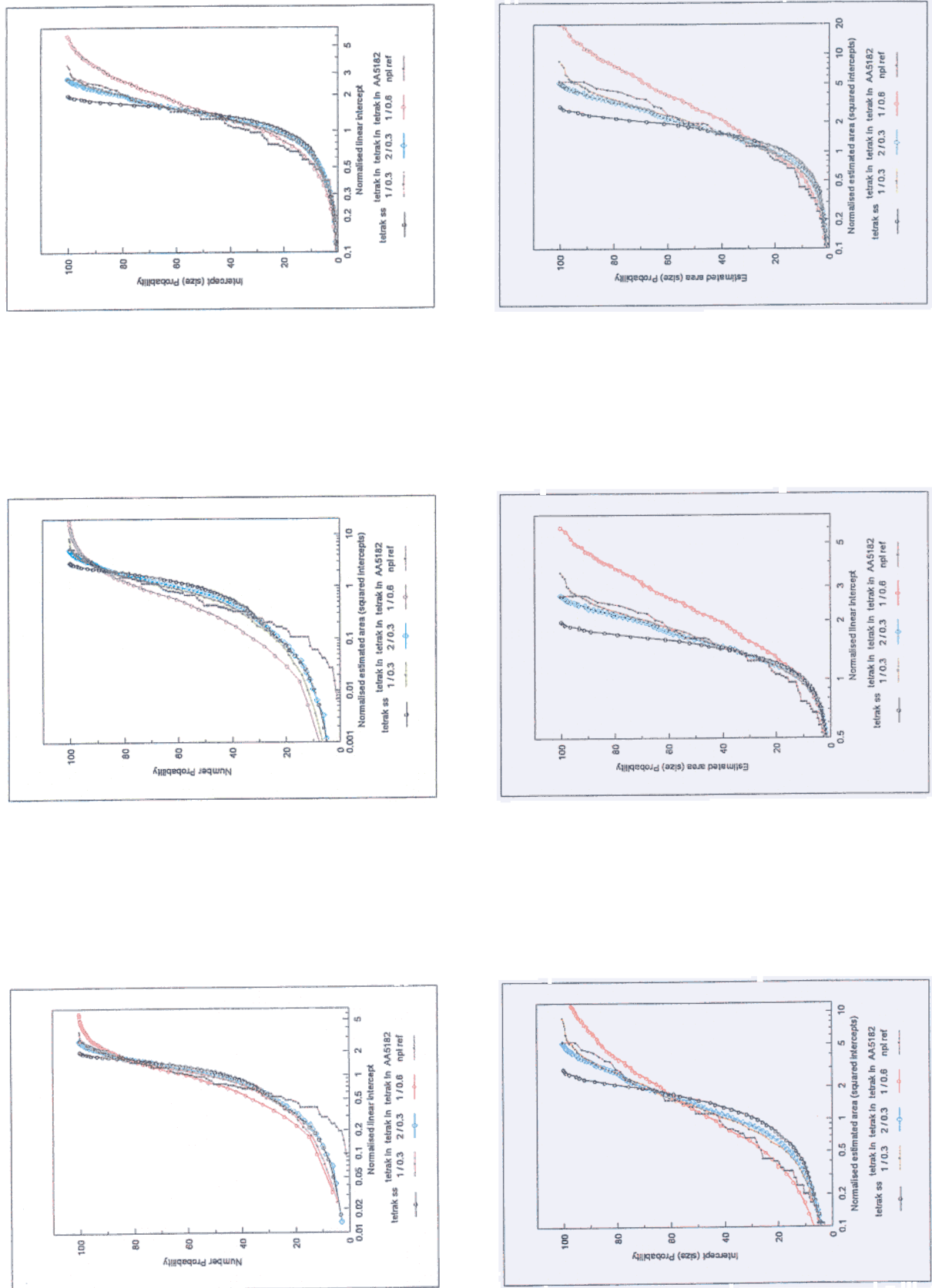


Fig 23 Comparison of distribution data from AA5182 and tetrakaidecahedron model.

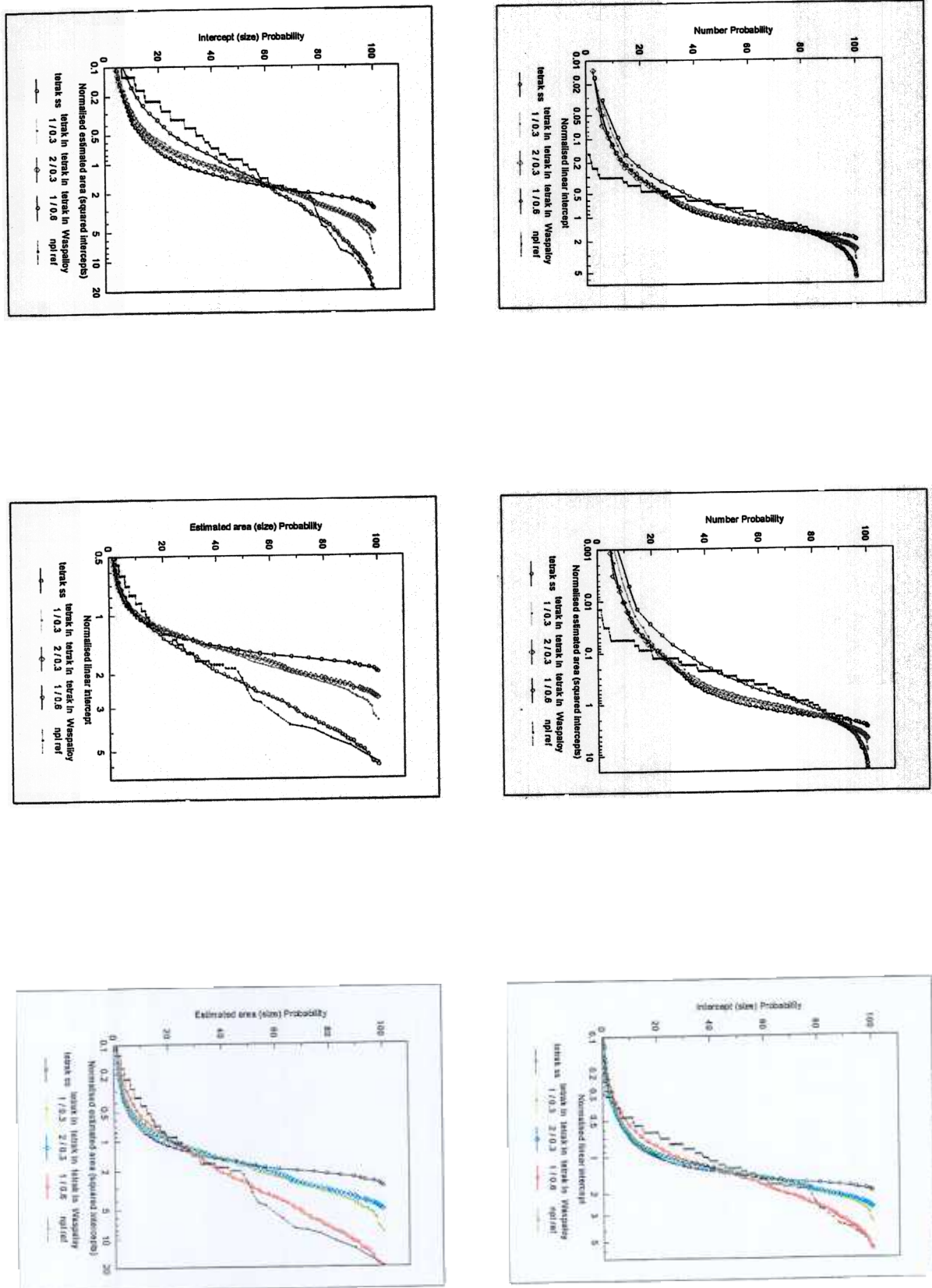


Fig 24 Comparison of distribution data from Waspaloy and tetrakaidecahedron model.

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APPENDIX B

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Clifton
Rugby CV23 0AR

APPENDIX C**MMP7 CONTACT LIST**

(Names additional to Appendix A and B. Some names in Appendix A and B are common to the MMP7 list but have been omitted here)

C Millward	Avesta
K Dicks	Oxford Instruments
T Golland	Quality Assessment
G Black	Sheffield Testing Laboratories
M Clarke	British Steel - Stocksbridge
P Bradbury	Buehler Krautkramer
M Deven	Buehler Krautkramer
G Gregory	CATRA
P Morris	British Steel - Swinden
J Rankin	Presto Eng Cutting Tools
P Hewlett	Pandrol UK Ltd
I Mitchell	J Sykes Brothers
S Smith	Lee Steel Strip
E White	Metallurgical & Inspection Services

MMP14 GRAIN SIZE MEASUREMENT FIRST STAGE, INTER - LABORATORY EXERCISE

The images contained in the pack are not included in this Appendix. Representative images of the 3 materials of the ILE are included in Figs 1-3 of the main text.

PACK CONTENTS

Please check that the contents of your Inter - Laboratory Exercise (ILE) stage I pack are as follows.

1) Printed Reference Images and Calibration Images

Image 1,	Calibration, x10 Objective.	
Image 2,	Waspaloy, x10 Objective	
Image 3,	Waspaloy, x10 Objective	(with reference intercept)
Image 4,	Calibration, x20 Objective	
Image 5,	IN909, x20 Objective	
Image 6,	IN909, x20 Objective	(with reference intercept)
Image 7,	Calibration, Macroviewer	
Image 8,	AA5182, Macroviewer	
Image 9,	AA5182, Macroviewer	(with reference intercept)

2) Two sets of floppy disks*, 6 disks in total. These contain the above images in digital format for those participants using automatic or semi - automatic image analysis systems. The disks do not contain the images with the reference intercepts. These images are 1024 x 772 pixels, stored as monochrome (grey) TIF files.

Set 1	DISK 1	Calib10.tif		775kB
	DISK 2	Wasp2b.tif	(Waspaloy)	775kB
	DISK 3	Calib20.tif		775kB
Set 2	DISK 4	In9091b.tif	(IN909)	775kB
	DISK 5	AlCal200.tif		775kB
	DISK 6	ALFIELD6.tif	(AA5182)	775 kB

3) Set of instructions for measurement of printed images plus results proforma for manual measurements, data sheets 1 to 6 and checklist.

The distance between adjacent scale divisions on the image is 10µm prior to magnification. Measure across the image starting at a left edge of a scale division and ending at a left edge of a scale division using as much of the image as possible. We recommend using a steel rule *. If you use an alternative please provide details on the check list. The measurement shall be made in mm. Count the number of scale divisions measured.

The magnification of the image is calculated as follows:-

Magnification = Length of line in mm X 100 / number of divisions

Record the calculated magnification on the results sheet 1.

- 6) Repeat the above operation for Image 4, Calibration X20 objective. This image is to be used for determining the magnification of Image 5 and Image 6, IN9091 x20 objective.

Record the calculated magnification on the results sheet 2.

- 8) Repeat the above operation for Image 7, Calibration, Macroviewer. This image is to be used for calculating the magnification of Image 8 and Image 9, AA5182, Macroviewer.

Record the calculated magnification on the results sheet 3.

- 4) Image 3, Waspaloy, x10 Objective contains a line drawn across the reference microstructure. Measure the lengths of the individual intercepts of this line with the underlying grains. Grains which touch the border of the image are not to be measured. The intercept lengths (in mm) are entered onto data sheet 1, as well as the calculated length of the intercept in µm. This is calculated by multiplying the intercept length in mm by 1,000 and dividing by the magnification obtained from Image 1.
- 5) From the measurements on data sheet 1, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 1.
- 6) Repeat steps 4) and 5) above for Image 6, IN9091, x20 Objective. Use the magnification obtained from Image 4 to calculate the linear intercepts and enter the data onto data sheet 2.

* * The steel rule should conform to BS 4372, 1968 (1996), Specification for Engineers Steel Measuring Rules

QY Results from page 3, page 1

[illegible]

Results from Image 9, Referenc

Dear Sir,

Mean Linear Intercept Grain Size, μm
Standard Deviation

at step 3) above. The linear intercept lines a
or equal to the arithmetic mean linear intercept
onto data sheet 10. Alternatively, a spreadsheet

If an "In - House" procedure exists for grain size measurement by semi or fully automatic image analysis, please use it on images Wasp2b.tif, In9091b.tif and ALFIELD6.tif. Please send details of the procedure and results on a separate sheet of paper.

ALTERNATIVE INSTRUCTIONS FOR PARTICIPANTS USING SCANNERS OR MACROVIEWERS

- 11) If a scanner or macroviewer is used to obtain an image for your image analysis system, please give additional information on the second checklist for data return. Ensure that the whole of the image is acquired, even if this means an empty border around part of the image. It is important that measurements can be made on the whole of the image provided rather than portions of it such that the statistics are not biased.
- 12) Use Image 1 and 2 instead of data files Calib10.tif and Wasp2b.tif. Follow the instructions as per steps 1, 2 and 3. Instead of using line 386 of the image, use the midpoint of the Y axis of your image for step 2..
- 13) Use Image 4 and 5 instead of data files Calib20.tif and In9091b.tif. Follow the instructions as per steps 4, 5 and 6. Instead of using line 386 of the image, use the midpoint of the Y axis of your image for step 5.
- 14) Use Image 7 and 8 instead of data files AlCal200.tif and ALFIELD6.tif. Follow the instructions as per steps 7, 8 and 9. Instead of using line 386 of the image, use the midpoint of the Y axis of your image for step 8.

'acto

W:WGSUN.LN/V1.7

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COMPANY			TICK BOX	SPREADSHEET NAME
CONTACT NAME				
DATA SHEET / SPREADSHEET	7	STEP 1 AND 2 (12)		
	8	STEP 3 (12)		
	9	STEP 4 AND 5 (13)		
	10	STEP 6 (13)		
	11	STEP 7 AND 8 (14)		
	12	STEP 9 (14)		
IN HOUSE PROCEDURE AND MEASUREMENTS		STEP 10		

**THE FOLLOWING SECTION IS FOR PARTICIPANTS USING SCANNERS OR
MACROVIEWERS**

	STEP 11	SIZE OF REFERENCE IMAGE IN PIXELS, NOT TOTAL SIZE OF IMAGE
WAS A SCANNER USED	YES / NO	
WAS A MACROVIEWER USED	YES / NO	

ADDITIONAL COMMENTS

Please add any additional information concerning the ILE, whether problems were encountered or where improvements could be made.

[illegible]

DATA SHEET 7.

Waspaloy image file Wasp2b.tif Results for line 386.

[illegible]

INSTRUCTIONS FOR DIGITAL IMAGES, TIF FIL

Six disks are supplied for those participants with access to automatic or semi - automatic image analysis systems, which can access stored digital images. For those who cannot use these digital images but can input images using a scanner or macroviewer, proceed to alternative instructions. The images on disk are the same as supplied in printed format except for two differences:-

- i) The reference intercept line as drawn on images 3, 6 and 9 are not drawn. These are at line 386 in the Y axis of the image.
 - ii) The boundary pixels have not been converted into a drawn border.
- 1) File Calib10.tif, DISK 1, (printed as Image 1) shall be used to calibrate the image analysis system for subsequent measurements on image file Wasp2b.tif (printed as Image 2). Enter the calibration or scaling factor obtained on data sheet 7. The factor obtained is usually expressed as $\mu\text{m} / \text{pixel}$. If a different type of factor is used by your system, please specify the units. Specify on the check list sheet if semi - automatic image analysis is being used, ie, the operator marks off the beginning and end points of the intercept lengths.
 - 2) Measure the linear intercepts along line 386 in the image, this corresponds to the reference line in the printed image. Please enter the data onto data sheet 7. Alternatively, a spreadsheet data file may be returned.
 - 3) Participants are to make their own linear intercept measurements in the X direction. The linear intercept lines are to be spaced at a distance greater than or equal to the arithmetic mean linear intercept determined from data sheet 4. Please enter the data onto data sheet 8 as well as the line position, ie, line 386 as per the reference line. Alternatively, a spreadsheet data file may be returned.
 - 4) File Calib20.tif, DISK 3 (printed as Image 4), shall be used to calibrate the image analysis system for subsequent measurements on image file In9091b.tif (printed as Image 5). Enter the calibration or scaling factor obtained on data sheet 9.
 - 5) Measure the linear intercepts along line 386 in the image, this corresponds to the reference line in the printed image. Please enter the data onto data sheet 9. Alternatively, a spreadsheet data file may be returned
 - 6) Repeat step 3) above. The linear intercept lines are to be spaced at a distance greater than or equal to the arithmetic mean linear intercept determined from data sheet 5. Enter data onto data sheet 10. Alternatively, a spreadsheet data file may be returned.
 - 7) File AlCal200.tif, DISK 5 (printed as Image 7), shall be used to calibrate the image analysis system for subsequent measurements on image file ALFIELD6.tif (printed as Image 8). Enter the calibration or scaling factor obtained on data sheet 11.
 - 8) Measure the linear intercepts along line 386 in the image, this corresponds to the reference line in the printed image. Please enter the data onto data sheet 11. Alternatively, a spreadsheet data file may be returned.

DATA SHEET 2.

IN909 Results from Image 6, Reference Intercept Line.

[illegible]

- 7) Repeat steps 4) and 5) above for Image 9, AA5182, Macroviewer. Use the magnification obtained from Image 7 to calculate the linear intercepts and enter the data onto data sheet 3.
- 8) Image 2 contains the same reference microstructure as Image 3, but does not contain the intercept line. Participants are to draw their own lines on the image for linear intercept measurements. These lines shall be drawn parallel to the X axis of the image and spaced apart such that any grain is not intercepted by more than one line. For each of these lines, the intercept of the line with the underlying grain is measured as described by step 4). Enter the results onto data sheet 4 ensuring that the correct magnification factor obtained from Image 1 is used to calculate the intercept length in μm . Please return the image with the drawn lines.
- 9) Repeat step 8) for Image 5 using the magnification factor obtained from Image 4. Enter the data onto data sheet 5.
- 10) Repeat step 8) for Image 8 using the magnification factor obtained from Image 7. Enter the data onto data sheet 6.
- 11) If an "In - House" procedure exists for grain size measurement, please use it on images 2, 5 and 8. Please send details of the procedure and results on separate sheets of paper.

Participants may return data in the form of spreadsheets if they wish.

Additional copies of the images are available if required.

- 4) Set of Instructions for measurement of digital images using image analysis systems plus results proforma, data sheets 7 to 12 and checklist
- 5) Additional blank data sheets for use as required.

If any of these items are missing or damaged, please contact me⁺ for replacements. If the images are required in a different computer file format, ie, JPEG, BMP, IMG, etc, please contact me for additional disks.

* All digital files were checked for viruses prior to copying to the disks.

⁺ E G Bennett, NPL.