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Round Robin on Grain Size Measurement for Advanced Technical Ceramics

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Abstract

The results of a joint CEN/VAMAS round robin on grain size measurement for advanced technical ceramics have been analysed. Twenty-five participants from Europe, USA and Japan employed a line and circle method for determination of the mean linear intercept length, and grid methods for determination of a grain size distribution. The results show that the scatter obtained for measurement of the mean linear intercept length and the grain size distribution is primarily due to the influence of micrograph preparation and interpretation and, to a lesser extent, also due to random positioning of lines, circles and grids. The results for the mean linear intercept length measurement validate the methods described in a proposed CEN standard. The grid methods for grain size distribution measurement seem suitable for implementation in future standards. Micrograph preparation and interpretation should be given special attention, as these factors are of major importance for consistent results.

Die Ergebnisse eines gemeinsamen CEN/VAMAS Vergleichstests zur Bestimmung der Korngröße an modernen technischen Keramiken wurden analysiert. Fünfundzwanzig Teilnehmer aus Europa, den USA und Japan benutzten eine Linien- und Kreismethode zur Bestimmung der mittleren linearen Schnittlänge und Rasterverfahren zur Bestimmung der Korngrößenverteilung. Die Ergebnisse zeigen, daß die Streuung der Meßwerte für die lineare Schnittlänge und die Korngrößenverteilung in erster Linie auf die Präparation und die Auswertung der Aufnahmen zurückzuführen ist, und nur in geringerem Maße auf die willkürlichen Positionen der Linien, Kreise und Raster. Die Ergebnisse der Schnittlängenbestimmung rechtfertigen die in einem CEN-Standard vorgeschlagenen und beschriebenen Methoden. Die Rasterverfahren zur Bestimmung der Korngrößenverteilung erscheinen zur Einführung in zukünftige Standards geeignet. Der Präparation und Auswertung der Aufnahmen sollten dabei besondere Aufmerksamkeit zuteil werden, da diese einen entscheidenden Einfluß auf die Konsistenz der Ergebnisse haben.

Les résultats d'une série d'essais interlaboratoires conjoints au CEN et au VAMAS sont analysés. Ils portent sur la mesure de la taille des grains de céramiques techniques de pointe. Vingt-cinq participants d'Europe, des Etats-Unis et du Japon ont utilisé la méthode de la ligne et du cercle pour déterminer la longueur moyenne linéaire d'interception, et les méthodes utilisant les grilles pour la détermination de la distribution granulométrique. Les résultats montrent que la dispersion obtenue pour la mesure de la longueur moyenne linéaire d'interception est d'abord due à l'influence de la technique de préparation et d'interprétation des micrographies et, dans une moindre mesure, aussi due au positionnement aléatoire des lignes, cercles et grilles. Les résultats obtenus pour la mesure de la longueur moyenne linéaire d'interception valident les méthodes décrites dans une proposition de norme CEN. Les méthodes utilisant une grille pour la mesure de la distribution granulométrique paraissent adaptées à leur mise en application dans de futures normes. La préparation et l'interprétation des micrographies devraient retenir spécialement l'attention, comme ces facteurs ont une importance indéniable pour obtenir des résultats cohérents.

1 Introduction and Objectives

Determination of the grain size of advanced technical ceramic materials is an important part of their characterization, both for material development and for the characterization of commercially available products. For this purpose it was agreed to

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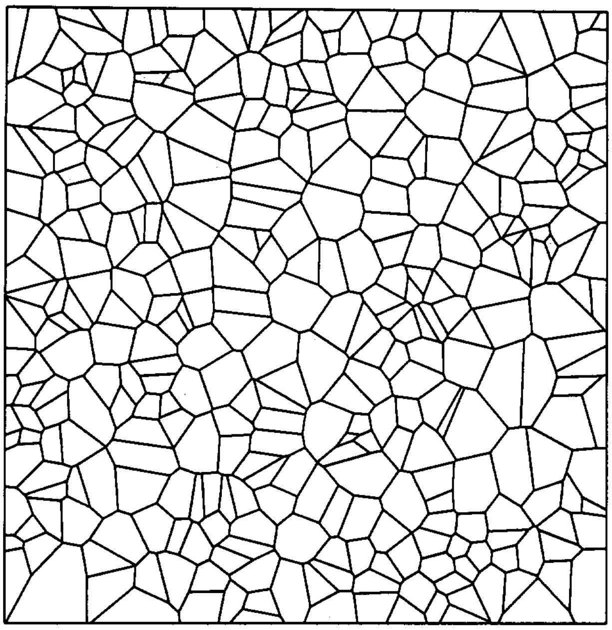

Fig. 1. Computer drawn microstructure (Dirichlet tesselation) with 350 grains. True dimensions 159 × 159 mm.

formulate a standard within the framework of the European normalization of advanced technical ceramics.[1] As such a standard for ceramic materials was proposed for the first time it seemed useful to examine its applicability by means of a round robin exercise. The round robin was carried out as a joint CEN/TC184 Working Group 3 and VAMAS TWA #3 exercise during 1992. The results of this round robin may give indications of the scatter of results between participants, the degree of difficulty experienced and possible uncertainties about the range of applicability.

The primary objective of the round robin was to test the reproducibility of results according to the draft standard for grain size measurement by the manual determination of the mean linear intercept length.[1] This was done in two ways. The first way uses an 'ideal' microstructure with no porosity and no second phase. This will determine the scatter of results between participants inherent in the random positioning of lines and circles on the 'micrograph', which therefore represents the minimum attainable scatter without significant error due to misinterpretation of grain boundaries or other features. The second way is more practical and is based on repeating the above procedure on a micrograph of a polished and thermally etched specimen. This will determine the additional scatter due to interpretation of a non-ideal microstructure and due to variation between areas randomly selected on specimens from a single batch of material. Two materials were used with strongly different grain size distributions as the actual grain size distribution is likely to be of importance for the level of scatter. The secondary objective of the round robin was to test a procedure for manually producing a grain size distribution which could be used in possible future standards.

2 Materials and Methods

The participants of the round robin received the draft CEN standard and a set of instructions and materials to complete the tasks given in the instructions.

Part 1 of the round robin dealt with the

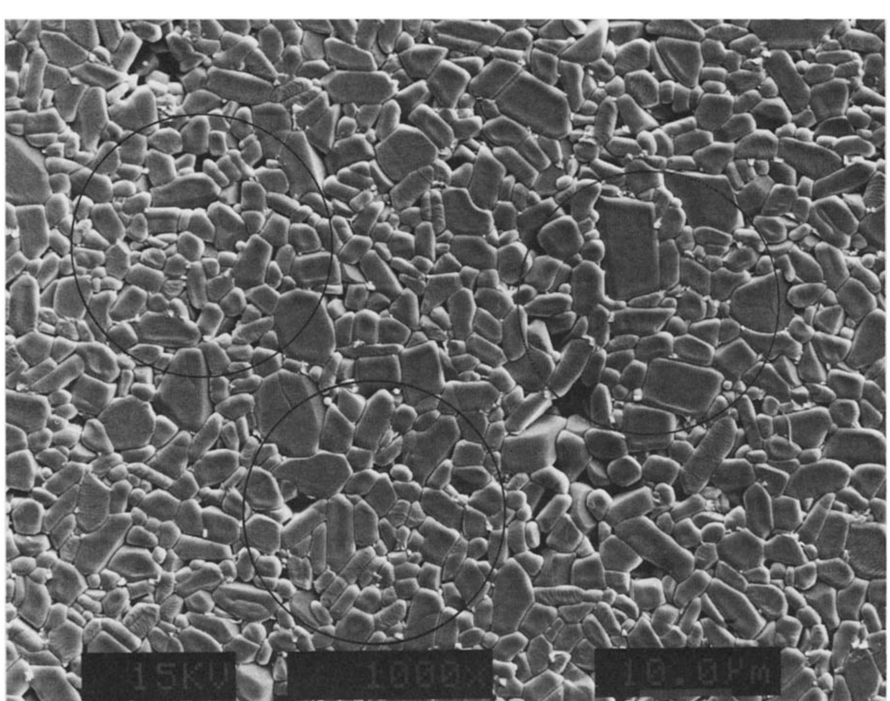

Fig. 2. SEM micrograph of sample of material 1 from a particular participant. Etching: 30 min at 1350°C.

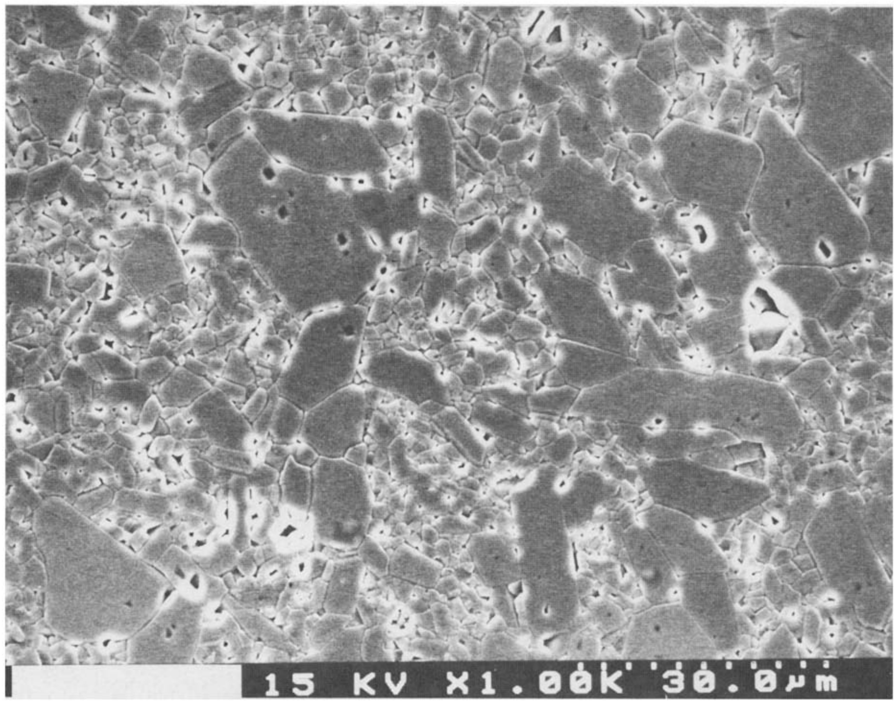

Fig. 3. SEM micrograph of material 2 used in Part 3 by all participants. True dimensions 242 × 176 mm.

determination of the mean intercept length by counting intersections of grains with a line or a circle method as described in the draft standard. For this purpose a computer-generated 'microstructure' was used, as given in Fig. 1. This microstructure was generated as a pseudo-random two-dimensional Dirichlet tesselation[2] containing 350 grains with no porosity.

Part 2 of the round robin also dealt with the determination of the mean intercept length by a line or circle method, but now using a micrograph made by the participant after polishing and etching of a supplied specimen of material 1 (96% brown alumina containing 4% submicron $MgTiO_3$ as second phase, sintered at about 1350°C). Figure 2 shows a micrograph made by scanning electron microscopy (SEM) of this material by one of the participants.

Part 3 of the round robin asked the participants to use a supplied micrograph of material 2 (99% alumina, sintered at about 1650°C), as shown in Fig. 3. This micrograph was to be used for measurement of the mean intercept length by a line and circle method and for determination of the grain size distribution. For the determination of a grain size distribution using a grid method two procedures were given as a guideline. Both essentially require that the distance between intercepts of grain boundaries and horizontal and vertical grid lines are measured. Method 1 used a forbidden line technique,[3] whereas in method 2 all fully visible grains on the micrograph were to be taken into consideration.

Part 4 of the round robin was identical to Part 3 except that now a specimen of material 2 was to be used by the participants to prepare a micrograph after polishing and etching. The resulting micrograph was used for measurement of mean intercept length and grain size distribution as already discussed.

3 Results

This section gives a compilation of the results of the round robin. More extended information can be found in the report produced for evaluation of the round robin and which was agreed upon by all participants.[4] Twenty-five participants sent results to the organisers after receiving instructions, materials and a reply form. Fourteen participants were from Europe, four from Japan and seven from the USA.

3.1 Part 1: Computer-drawn microstructure

The results for Part 1 consist of values for the total line length (line method) or circle circumference (circle method) L, the total number of intersections n_i and the mean linear intercept length l given by $l = L/n_i$. Characteristic data obtained for the line and circle method are given in Table 1, where the participants used 3–5 lines and circles for the analyses as proposed in the draft CEN standard. An overview of the results for the line method is given in Fig. 4, while for the circle method a similar picture applies.

From these data, using the Welsh–Aspin T-test on the average values for l,[5] it is found that the line and circle methods do not yield statistically different

Table 1. Characteristic results for Part 1: computer drawn microstructure with mean linear intercept length measurement by line and circle methods

	Line method	*Circle method*
Number of results, N	25	25
Average L (mm)	703	869
Average n_i	100	125
Average l (mm)	7·01	6·96
Sample standard deviation for average l (mm)	0·41	0·29
95% confidence interval for average l (mm) (t-distribution with $N-1$ degrees of freedom)	6·84–7·18	6·84–7·08

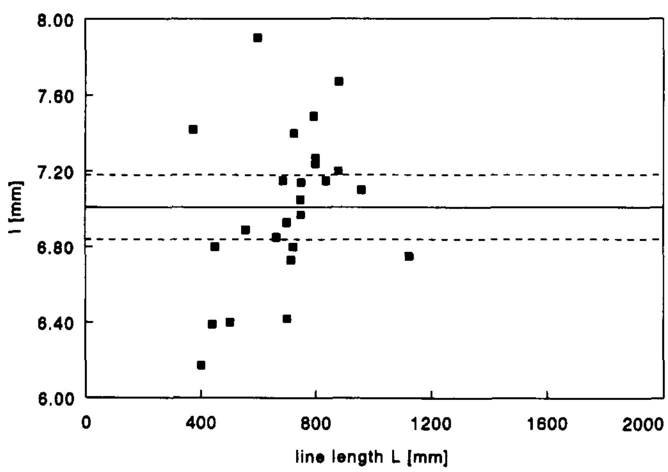

Fig. 4. Mean linear intercept length l as a function of the line length L in Part 1. ——, Average value for l; — — —, 95% confidence interval.

mean linear intercept lengths (>95% confidence) and may therefore be considered identical. From the results obtained (Fig. 4), it was seen that in this part of the round robin some results have been reported which clearly lie outside the 95% confidence intervals. One obvious reason was found: most participants who used clearly less than 100 intersections for line or circle methods obtained a mean intercept length clearly outside the 95% confidence interval. Therefore it may be concluded that at least 100 intersections are required to obtain a reasonable approximation for l. One participant found a high value for l using the circle method. The reason for this may be that use was made of three concentric circles whereas in the draft CEN standard circles with random positioning of the centres are proposed, as use of concentric circles is believed to yield biased estimates.

In summary, it may be concluded that the line and circle methods for determination of the mean linear intercept length yielded the same results. It also was shown that the amount of scatter, defined as 2 × standard deviation/mean value, between various participants, is less than 10%, provided that at least 100 grain intersections with proper random positioning of lines and circles is used. This is considered as a reasonable value and may as a first indication be taken equal to $100\%/\sqrt{n_i}$. This scatter is likely to be due to the influence of random positioning of lines and circles on the micrograph. This scatter can be interpreted as the minimum attainable scatter in these types of measurements, as scatter due to misinterpretation of grain boundaries or other features is minimal.

3.2 Part 2: Sample of material 1

Part 2 of the round robin also dealt with the determination of the mean intercept length by the line and circle method. For both line and circle methods, characteristic results are given in Table 2, while Fig. 5 shows the detailed results for the case of the circle method. From these data it is readily found that again the line and circle methods do not yield statistically different mean linear intercept lengths (>95% confidence) and may therefore be considered identical.

From these results, it is seen that the scatter in the

Table 2. Characteristic results for Part 2: sample of material 1 with mean linear intercept length measurement by line and circle methods

	Line method	*Circle method*
Number of results, N	25	25
Average L (μm)	323	376
Average n_i	128	144
Average l (μm)	2·54	2·61
Sample standard deviation for average l (μm)	0·35	0·34
95% confidence interval for average l (μm) (t-distribution with $N-1$ degrees of freedom)	2·40–2·68	2·47–2·75

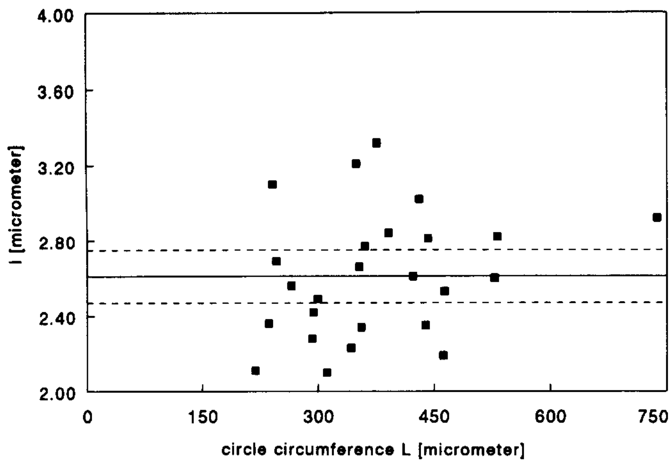

Fig. 5. Mean linear intercept length l of material 1 as a function of the circle circumference L in Part 2. ———, Average value for l; – – – –, 95% confidence interval.

results is about 25%, which is 15% higher than for Part 1. The reason for this is not so obvious, as only three participants used less than 100 grain intersections, which plays a disturbing role, as discussed in Section 3·1. Again, the more extreme result of one participant for the circle method is likely to be due to the use of concentric circles. It might therefore be expected that this increase in scatter is due to a combination of variations in preparation techniques, material properties between the distributed specimen (although taken from one batch) and different interpretation of features on a more complex micrograph. However, some systematic influence can be found if the etching temperatures and times reported are also taken into consideration. From these data it could be deduced (with some caution because of the limited amount of data for certain combinations) that the mean intercept length increases with increasing etching temperature and time. This observation becomes rather clear when considering the values for the line method for 30 min etching where etching temperatures of 1100, 1200, 1300, 1350 and 1400°C yielded an averaged intercept length l of 2·09, 2·38, 2·20, 2·58 and 2·94 μm, respectively. This need not necessarily be due to grain growth, although given a sintering temperature of about 1350°C this cannot be excluded. A too strong etching procedure may result in changes in the shape of the grains, e.g. rounding of corners and edges or in obscuration of small grains with increasing severity of thermal etch. This could be observed on many of the micrographs etched at higher temperatures. It is difficult, however, to estimate the precise influence of these factors on the scatter found, as for the results of eight specimens etched at 1350°C, 30 min, a scatter of 25% is still observed (sample standard deviation approximately 0·3 μm).

Some other comments on the results can also be made. Most of the micrographs were made using a scanning electron microscope (SEM), but some participants used an optical microscope (OM). Whether this has a systematic influence cannot be determined. It is worthwhile to note that the OM micrographs have a quite different appearance to those taken by SEM in the sense that grain boundaries are less pronounced. This is possibly due to levels of contrast introduced by the grains, which does not occur in the SEM micrographs. Some of the scatter in the results may also be due to the fact that the micrographs made by SEM have not been made with the same set-up of the SEM (working distance and voltage, for example). These differences may lead to a different interpretation of the microstructure. It should be mentioned that most participants carried out a calibration of the magnification of the SEM or optical microscope using certified graticules or a calibrated grating. In general the deviations from uniformity of magnification for the horizontal and vertical screen direction were limited, typically less than 1%.

At this moment it can be stated that in principle the methods for mean linear intercept length measurement agree well. The main causes for the scatter in the results are the influence of random positioning of lines and circles (as analysed in Part 1), the influence of etching temperature and time through possible grain growth and change of shape of the grains, the influence of the polishing/etching procedure, the selection of the area to examine and the set-up of the SEM or OM to make a micrograph where a certain amount of subjectivity is introduced. The latter three causes only are related to micrograph preparation and may obscure whether additional scatter is introduced due to the interpretation of a more complicated micrograph than the one used in Part 1. On the other hand it can be stated that material 1 had a 'nice' microstructure (equiaxed grains of relatively uniform size with clearly marked grain boundaries) and did not easily give rise to grain pluck-out so that the interpretation should not have been too complicated and should not have led to substantial additional scatter. Therefore it is concluded that the 15% increase in scatter could be attributed to the influence of micrograph preparation only.

3.3 Part 3: Micrograph of material 2

In Part 3 a supplied micrograph was to be used for measurement of the mean linear intercept length by the line and circle methods and for determination of the grain size distribution by means of a grid method.

3.3.1 Determination of mean linear intercept length

Mean linear intercept length characteristic results for material 2 are given in Table 3. From these data it

Table 3. Characteristic results for Part 3: micrograph of material 2 with mean linear intercept length measurement by line and circle methods

	Line method	*Circle method*
Number of results, N	25	24
Average L (mm)	758	928
Average n_i	135	173
Average l (mm)	5·70	5·48
Sample standard deviation for average l (mm)	0·73	0·75
95% confidence interval for average l (mm) (t-distribution with $N-1$ degrees of freedom)	5·39–6·00	5·17–5·79

is concluded that again the line and circle methods do not yield statistically different mean linear intercept lengths (>95% confidence with Welsh–Aspin T-test). As was concluded in Parts 1 and 2, those results where less than 100 grain intersections were counted or concentric circles were used were considered to be biased. With these results excluded, the scatter between the participants was about 20%, which is 10% higher than for Part 1 and 5% less than for Part 2. Because in this case all participants used the same micrograph, so that the disturbing influences of specimen preparation can be neglected, the increase in scatter with respect to Part 1 and Part 2 is likely to be due to the use of a microstructure with a wider grain size distribution than used in Part 2. Also the increased difficulties in evaluation of a 'real' micrograph from a material with a more complicated microstructure, where misinterpretation of grain boundaries and other features is possible, may have led to an increase in the scatter. The latter reason would also explain the increase in scatter with respect to Part 2 (if the larger part of the scatter in Part 2 is devoted to the influence of micrograph preparation) as the microstructure of material 1 is 'nicer' and therefore less eligible to misinterpretation.

3.3.2 Determination of grain size distribution

For the two methods used to determine a grain size distribution the results obtained by the participants consisted of measured intercept lengths between grain boundaries along grid lines. These data were returned on a floppy disk to the organizers and subsequently processed. Evaluation of the data showed that the data can be described well by means of a unimodal lognormal distribution with 50% probability grain size x_{50} and variance σ_{lnx}, yielding a correlation coefficient >0·99. The parameters x_{50} and σ_{lnx} for each participant were obtained from a linear curve fit on the cumulative distribution function plot on a lognormal scale using a division of grain size into ten classes with equidistant logarithmic spacing. An example of such a plot is shown in Fig. 6, while in Table 4 the results obtained are summarized. Figure 7 shows the values for x_{50} obtained as a function of the number of grid intersections for each participant for method 1 (forbidden line technique).

The scatter in x_{50} is of the same order of magnitude (25%) as was found for the mean linear intercept length with the line and circle methods. It is worthwhile mentioning that most of the participants used a grid of 15 × 15 to 20 × 20 mm.

Participants who used a much finer grid obtained relatively large values for x_{50}. This dependence on the grid size is not unexpected as for a small grid, large grains are likely to be intersected more often and therefore the mean grain size will increase. In the case of a coarse grid the question arises if sufficient intersections are present to have reliable statistics. This question cannot be answered at this moment because, as is illustrated in Fig. 7, no obvious relation between number of intersections and grain size is found. The grid dimensions of 15 × 15 to 20 × 20 mm are such that in general a grid of about 10 × 10 lines could be drawn on the micrograph as asked in the instructions: 'select the spacing of the

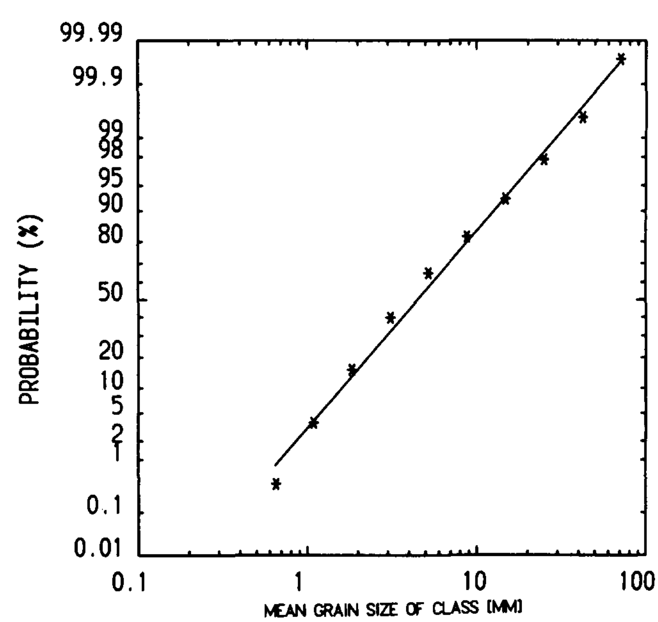

Fig. 6. Example of a cumulative frequency distribution obtained in Part 3 and plotted on a lognormal scale. Straight line represents least squares fit.

Table 4. Characteristic results for Part 3: micrograph of material 2 with grain size distribution measurement by grid methods 1 and 2

	Method 1	*Method 2*
Number of results, N	22	10
Average M	404	517
Average x_{50} (mm)	4·4	4·3
Sample standard deviation for average x_{50} (mm)	0·67	0·76
95% confidence interval for average x_{50} (mm) (t-distribution with $N-1$ degrees of freedom)	4·10–4·70	3·73–4·82

M = Number of grid intersections.

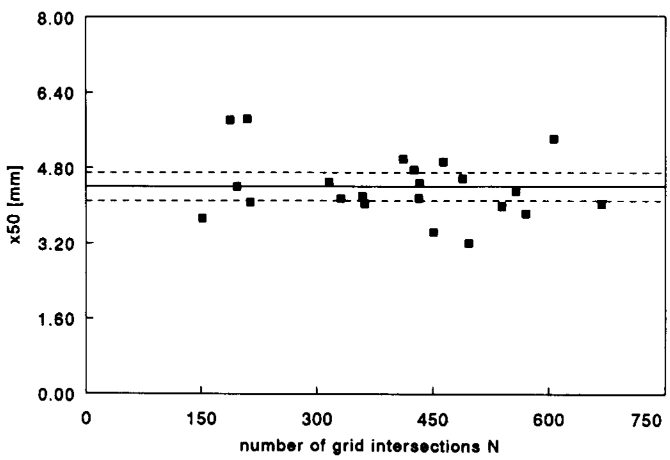

Fig. 7. Computed values for 50% probability grain size x_{50} of material 2 as a function of the number of grid intersections N for method 1 in Part 3. ———, Average value for x_{50}; — — —, 95% confidence interval.

grid to be approximately the same size as the largest grains so that each of these are not normally counted twice'. Here it was not meant to use the single largest grain on the micrograph, but a 'typical' large grain which was about 20 mm in size. The requirement for a 10×10 grid was given by the need to have sufficient grid intersections for statistical reliability, and this seems to have been sufficient.

No obvious difference between method 1 and method 2 is observed, although the number of results for method 2 is too small to give a definite answer. The differences that are expected to occur between these methods are therefore likely to be of less importance in practice where other factors, such as the influence of random positioning of the grid, the grid size and uniformity of the microstructure, have a larger impact on the variability of the results.

As a comparable analysis for grain size distribution measurement on ceramic materials is not available, to the organizers' knowledge, it is difficult to give an appreciation for this scatter. Given the consistency with the scatter in the mean linear intercept length and the fact that the microstructure of this material is not ideal, it is felt that the procedures defined in this round robin for grain size distribution measurement are suitable for implementation in future standards.

3.4 Part 4: Sample of material 2

In Part 4 a sample of material 2 was used by the participants to prepare a micrograph after polishing and etching. The resulting micrograph was used for measurement of mean linear intercept length and grain size distribution as discussed in the previous sections.

3.4.1 Determination of mean linear intercept length

Characteristic results for the determination of the mean linear intercept length are given in Table 5. Again no statistically significant differences between the line and circle methods are found. The scatter between the participants is about 35%, which is higher than for Part 1, Part 2 and Part 3. Some of the factors introducing the scatter found for Part 1, Part 2 and Part 3 have to be examined here. Firstly, the scatter of 10% due to random positioning of lines and circles found in Part 1 will inevitably play a role here too. Secondly, because the micrograph was prepared by the participants themselves possibly 15% scatter can be introduced through the preparation procedure, as was shown in Part 2. The preparation procedure was different for each participant, although in this case no obvious influence of etching temperature and time could be found. However, the preparation of a quality micrograph of material 2 proved to be more difficult, which was confirmed by remarks of some of the participants. In comparison with other participants, some micrographs seem to contain a relatively large number of large or small grains, which might explain the relatively high and low values, respectively, for the mean linear intercept length. Thirdly, as observed in Part 3, 10% scatter may be introduced due to the interpretation of a micrograph with a more complicated structure. If the three sources of scatter mentioned are added the total amounts about 35%, which would explain the scatter found here.

3.4.2 Determination of grain size distribution

As was done in Part 3, the grain size distribution was also determined in Part 4 using the two grid

Table 5. Characteristic results for Part 4: sample of material 2 with mean linear intercept length measurement by line and circle methods

	Line method	*Circle method*
Number of results, N	24	22
Average L (μm)	388	437
Average n_i	138	155
Average l (μm)	2·80	2·83
Sample standard deviation for average l (μm)	0·50	0·50
95% confidence interval for average l (μm) (t-distribution with $N-1$ degrees of freedom)	2·59–3·01	2·61–3·05

Table 6. Characteristic results for Part 4: micrograph of material 2 with grain size distribution measurement by grid methods 1 and 2

	Method 1	*Method 2*
Number of results, N	19	9
Average M	291	469
Average x_{50} (μm)	2·3	2·2
Sample standard deviation for average x_{50} (μm)	0·55	0·45
95% confidence interval for average x_{50} (μm) (t-distribution with $N-1$ degrees of freedom)	2·03–2·57	1·85–2·55

M = Number of grid intersections.

methods. Again evaluation of the data showed that they can be described well by means of a unimodal lognormal distribution. Table 6 summarizes the results obtained from the curve fits. Again, possible differences between method 1 and method 2 are difficult to be determined. The scatter in x_{50} is somewhat higher, 40–50%, than for the line and circles methods. From the discussion on the results for the mean linear intercept length measurement, it will also be clear that some of the results for the grain size distribution measurement will be more extreme because of the quality of the micrograph or because a relatively large number of small or large grains were present in the micrograph. Also, as was discussed for Part 3, the size of the grid may have its influence. Most of the other participants used a grid of 10 × 10 or 20 × 20 mm, but a much smaller grid is likely to lead to an increase in the mean grain size. The grain size distributions found are subjected to more scatter (40–50%) than those found in Part 3 for the same material (30%) where each participant used an identical micrograph. This increase of 10–20% will therefore be due to a combination of scatter introduced due to micrograph preparation, to selection of observed area, and scatter due to interpretation of the micrograph.

4 Conclusions and Recommendations

The results of a round robin exercise for grain size measurement of advanced technical ceramics have been discussed and analysed. Prime targets of this round robin were to validate a draft CEN standard for mean linear intercept length measurement and to test methods for manual determination of grain size distribution. Twenty-five companies, institutes and universities from Europe, Japan and the USA participated.

With respect to the line and circle methods proposed in the draft CEN standard for grain size measurement, it can be concluded that in principle these methods yield identical results. Some precautions with respect to the number of grain intersections and the randomness of lines and circles is necessary, however. Using about 100 grain intersections gives a scatter of about 10% which is considered as a reasonable number. Additional scatter can be introduced, however, depending on micrograph preparation and the degree of complexity of the microstructure. From the results given it may be concluded that a scatter of about 25% is likely to be found. Whether this is an acceptable number will depend on the actual application. The influence of micrograph preparation and micrograph interpretation must be considered as key factors. Reducing scatter by minimizing their influence requires experience and knowledge, which unfortunately cannot be standardized.

The second target of this round robin was to test methods for measurement of grain size distribution. The grid methods for grain size distribution measurement seem to yield relatively consistent results. Expected differences between method 1 and method 2 are not found, so they are likely to be of less influence than factors such as micrograph preparation, for example. The amount of scatter in the results is in general comparable to the scatter found for the measurement of mean linear intercept length. As such no objection seems to exist to the methods proposed.

From the results discussed it can be concluded that micrograph preparation and interpretation, which are not necessarily independent factors, are of major importance in determining the degree of scatter. The large scatter for Part 3 was possibly due to the fact that the micrograph provided is not of an optimal quality, which was done on purpose by the organizers. But the even larger scatter in Part 4 again shows the influence of micrograph preparation. From the assembled collection of micrographs returned by the participants striking differences in magnification, contrast, amount of reflections on SEM images, etc., are found. It is therefore suggested that some typical 'good' and 'bad' images are incorporated in the draft CEN standard as a guideline for users. Also it is believed that the influence of the etching procedure should be more specifically addressed, as it is likely to have a major influence on the micrograph quality. This also applies to details of the set-up of optical and SEM microscopes.

The methods discussed in this report in principle can also be used for determination of phase content and grain size distribution of multiphase materials. Whether this yields comparable results is uncertain. It would be well worthwhile to make this a topic for a future VAMAS or joint CEN/VAMAS round robin exercise to provide information useful for formulation of standards on these important topics.

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