LENGTH WEIGHTED GEOMETRIC MEAN DIAMETER OF FIBRES

1. **METHOD**

1.1 INTRODUCTION

This method describes a procedure to measure the Length Weighted Geometric Mean Diameter (LWGMD) of bulk Man Made Mineral Fibres (MMMF). As the LWGMD of the population will have a 95 % probability of being between the 95 % confidence levels (LWGMD \pm two standard errors) of the sample, the value reported (the test value) will be the lower 95 % confidence limit of the sample (i.e. LWGMD - 2 standard errors). The method is based on an update (June 1994) of a draft HSE industry procedure agreed at a meeting between ECFIA and HSE at Chester on 26/9/93 and developed for and from a second inter-laboratory trial (1, 2). This measurement method can be used to characterise the fibre diameter of bulk substances or products containing MMMFs including refractory ceramic fibres (RCF), man-made vitreous fibres (MMVF), crystalline and polycrystalline fibres.

Length weighting is a means of compensating for the effect on the diameter distribution caused by the breakage of long fibres when sampling or handling the material. Geometric statistics (geometric mean) are used to measure the size distribution of MMMF diameters because these diameters usually have size distributions that approximate to log normal.

Measuring length as well as diameter is both tedious and time consuming but, if only those fibres that touch an infinitely thin line on a SEM field of view are measured, then the probability of selecting a given fibre is proportional to its length. As this takes care of the length in the length weighting calculations, the only measurement required is the diameter and the LWGMD-2SE can be calculated as described.

1.2 DEFINITIONS

Particle: An object with a length to width ratio of less than 3:1.

Fibre: An object with a length to with ratio (aspect ratio) of at least 3:1.

REFERENCE SUBSTANCES

None

PRINCIPLE OF THE TEST METHOD

A number of representative core samples are taken from the fibre blanket or from loose bulk fibre. The bulk fibres are reduced in length using a crushing procedure and a representative sub-sample dispersed in water. Aliquots are extracted and filtered through a 0.2 μ m pore size, polycarbonate filter and prepared for examination using scanning electron microscope (SEM) techniques. The fibre diameters are measured at a screen magnification of x10,000 {this was for 3µm fibres, for 6µm fibres 5,000 may be more suitable} or greater using a line intercept method to give an unbiased estimate of the median diameter. The lower 95% confidence interval (based on a one sided test) is calculated to give an estimate of the lowest value of the geometric mean fibre diameter of the material.

Scope and Limitations

The method is designed to look at diameter distributions which have median diameters from 0.5 μ m to 6 μ m. Larger diameters can be measured by using lower SEM magnifications but the method will be increasingly limited for finer fibre distributions and a TEM (transmission electron microscope) measurement is recommended if the median diameter is below 0.5 μ m.



QUALITY CRITERIA

Not stated

DESCRIPTION OF THE TEST METHOD

Safety/precautions

Personal exposure to airborne fibres should be minimised and a fume cupboard or glove box should be used for handling the dry fibres. Periodic personal exposure monitoring should be carried out to determine the effectiveness of the control methods . When handling MMMF's disposable gloves should be worn to reduce skin irritation and to prevent cross-contamination.

Apparatus / equipment

- Press and dyes (capable of producing 10 MPa).
- 0.2 µm pore size polycarbonate capillary pore filters (25 mm diameter).
- 5 µm pore size cellulose ester membrane filter for use as a backing filter.
- Glass filtration apparatus (or disposable filtration systems) to take 25 mm diameter filters (e.g. Millipore glass microanalysis kit, type no XX10 025 00).
- Freshly distilled water that has been filtered through a 0.2 µm pore size filter to remove micro-organisms.
- Sputter coater with a gold or gold/palladium target.
- Scanning electron microscope capable of resolving down to 10 nm and operating at x10,000 magnification.
- Miscellaneous: spatulas, type 24 scalpel blade, tweezers, SEM tubes, carbon glue or carbon adhesive tape, silver dag.
- Ultrasonic probe or bench top ultrasonic bath
- Core sampler or cork borer, for taking core samples from MMMF blanket

Test Procedure

Sampling

For blankets and bats a 25 mm core sampler or cork borer is used to take samples of the cross-section. These should be equally spaced across the width a of small length of the blanket or taken from random areas if long lengths of the blanket are available. The same equipment can be used to extract random samples from loose fibre. Six samples should be taken when possible, to reflect spatial variations in the bulk material.

The six core samples should be crushed in a 50 mm diameter die at 10 MPa. The material is mixed with spatula and repressed at 10 MPa. The material is then removed from the die and stored in a sealed glass bottle.

Sample Preparation

If necessary, organic binder can be removed by placing the fibre inside a furnace at 450 °C for about one hour. Cone and quarter to subdivide the sample (this should be done inside a dust cupboard).

Using a spatula, add a small amount (<0.5 g) of sample to 100 ml of freshly distilled water that has been filtered through a 0.2 μ m membrane filter (alternative sources of ultra pure water may be used if they are shown to be satisfactory). Disperse thoroughly by the use of an ultrasonic probe operated at 100 W power and tuned so that cavitation occurs. (If a probe is not available use the following method: repeatedly shake and invert for 30 seconds; ultrasonic in a bench top ultrasonic bath for five minutes; then repeatedly shake and invert for a further 30 seconds).

Immediately after dispersion of the fibre, remove a number of aliquots (e.g. three aliquots of 3, 6 and 10 ml) using a wide-mouthed pipette (2-5 ml capacity).

Vacuum filter each aliquot through a 0.2 μ m polycarbonate filter supported by a 5 μ m pore MEC backing filter, using a 25 mm glass filter funnel with a cylindrical reservoir. Approximately 5 ml of filtered distilled water should be placed into the funnel and the aliquot slowly pipetted into the water holding the pipette tip below the meniscus. The pipette and the reservoir must be flushed thoroughly after, pipetting as thin fibres have a tendency to be located more on the surface.



Carefully remove the filter and separate it from the backing filter before placing it in a container to dry.

Cut a quarter or half filter section of the filtered deposit with a type 24 scalpel blade using a rocking action. Carefully attach the cut section to a SEM stub using a sticky carbon tab or carbon glue. Silver dag should be applied in at least three positions to improve the electrical contact at the edges of the filter and the stub. When the glue / silver dag is dry sputter coat approximately 50 nm of gold or gold/palladium onto the surface of the deposit.

SEM calibration and operation

Calibration

The SEM calibration should be checked at least once a week (ideally once a day) using a certified calibration grid. The calibration should be checked against a certified standard and if the measured value (SEM) is not within $\pm 2\%$ of the certified value, then the SEM calibration must be adjusted and re-checked.

The SEM should be capable of resolving at least a minimum visible diameter of $0.2 \mu m$, using a real sample matrix, at a magnification of x 2,000.

Operation

The SEM should be operated at 5.000 magnification using conditions that give good resolution with an acceptable image at slow scan rates of, for example, 5 seconds per frame. Although the operational requirements of different SEMs may vary, generally to obtain the best visibility and resolution, with relatively low atomic weight materials, accelerating voltages of 5 – 10 keV should be used with a small spot size setting and short working distance. As a linear traverse is being conducted, then a 0° tilt should be used to minimise re-focussing or, if the SEM has a eucentric stage, the eucentric working distance should be used. Lower magnification may be used if the material does not contain small (diameter) fibres and the fibre diameters are large (>5µm)

Sizing

Low magnification examination to assess the sample

Initially the sample should be examined at low magnification to look for evidence of clumping of large fibres and to assess the fibre density. In the event of excessive clumping it is recommended that a new sample is prepared. For statistical accuracy it is necessary to measure a minimum number of fibres and high fibre density may seem desirable as examining empty fields is time consuming and does not contribute to the analysis. However, if the filter is overloaded, it becomes difficult to measure all the measurable fibres and, because small fibres may be obscured by larger ones, they may be missed.

Bias towards over estimating the LWGMD may result from fibre densities in excess of 150 fibres per millimetre of linear traverse. On the other hand, low fibre concentrations will increase the time of analysis and it is often cost effective to prepare a sample with a fibre density closer to the optimum than to persist with counts on low concentration filters. The optimum fibre density should give an average of about one or two countable fibre per fields of view at 5,000 magnification. Nevertheless the optimum density will depend on the size (diameter) of the fibres, so it is necessary that the operator uses some expert judgement in order to decide whether the fibre density is close to optimal or not.

Length weighting of the fibre diameters

Only those fibres that touch (or cross) an (infinitely) thin line drawn on the screen of the SEM are counted. For this reason a horizontal (or vertical) line is drawn across the centre of the screen.

Alternatively a single point is placed at the centre of the screen and a continuous scan in one direction across the filter is initiated. Each fibre of aspect ratio grater than 3:1 touching or crossing this point has its diameter measured and recorded.

Fibre sizing

It is recommended that a minimum of 300 fibres are measured. Each fibre is measured only once at the point of intersection with the line or point drawn on the image (or close to the point of intersection if the fibre edges are

obscured). If fibres with non-uniform cross sections are encountered, a measurement representing the average diameter of the fibre should be used. Care should be taken in defining the edge and measuring the shortest distance between the fibre edges. Sizing may be done on-line, or off-line on stored images or photographs. Semi-automated image measurement systems that download data directly into a spreadsheet are recommended, as they save time, eliminate transcription errors and calculations can be automated.

The ends of long fibres should be checked at low magnification to ensure that they do not curl back into the measurement field of view and are only measured once.

2 DATA

2.1 TREATMENT OF RESULTS

Fibre diameters do not usually have a normal distribution. However, by performing a log transformation it is possible to obtain a distribution that approximates to normal.

Calculate the arithmetic mean (lnD) and standard deviation (SD_{lnD}) of the lnD (log to base e) values of the n fibre diameters (D).

$$\overline{\ln D} = \frac{\sum \ln D}{n}$$
(1)
$$\ln D = \sqrt{\frac{\sum \left(\ln D - \overline{\ln D} \right)^2}{n-1}}$$
(2)

The standard deviation is divided by the square root of the number of measurements (n) to obtain the standard error (SE_{inD}) .

SD

$$SE_{lnD} = \frac{SD}{\sqrt{n}}$$
 (3)

Substract two times the standard error from the mean and calculate the exponential of this value (mean minus two standard error) to give the geometric mean minus two geometric standard errors.

$$LWGMD - 2SE = e^{\overline{\ln D} - 2SE_{\overline{\ln D}}}$$
(4)

REPORTING

3

TEST REPORT

The test report should include at least the following information:

- The value of LWGMD-2SE.
- Any deviations and particularly those which may have an effect on the precision or accuracy of the results with appropriate justifications

4 **REFERENCES**

1. B. Tylee SOP MF 240. Health and Safety Executive. February 1999.

2. G. Burdett and G. Revell. Development of a standard method to measure the length-weighted geometric mean fibre diameter: Results of the Second inter-laboratory exchange. IR/L/MF/94/07. Project R42.75 HPD. Health and Safety Executive. Research and Laboratory Services Division. 1994.