

Reliable Determination of Particle Size in Hardmetal Powders in the Sub-Micrometre Range

Proposal for an investigation under the aegis of the European Powder Metallurgy Association, European Hard Materials Group

Background

Established hardmetal products are available with carbide grain sizes down to about 0.3µm and there is a growing interest in the use of materials with average grain sizes down to at least 0.1µm because of the increasing hardness that results from this together with acceptable toughness. Manufacture of such products necessitates using powders which are equally fine or even finer. To understand the behaviour of such powders during processing and to control the quality of the resulting products requires accurate and reproducible characterisation of the starting materials in terms of their particle size distributions. However, it is clear that several of the methods that are adopted for sizing conventional powders are not capable of application in the sub-micron range. A recent review of possible techniques for characterising size distributions of such fine powders concluded that the following methods could be suitable:

- High resolution scanning electron microscopy (FEG-SEM)
- Gas adsorption (BET)
- Photon correlation spectroscopy (PCS)
- Centrifugal sedimentation
- Acoustic attenuation spectroscopy (AAS)

Aims

The relative strengths and weaknesses of the different approaches listed above, as applied to hardmetal powders, are not evident at present. In order to satisfy the needs of suppliers and customers in the industry, the most suitable technique(s) should be evaluated with regard to:

- Accuracy
- Precision/reliability
- Convenience
- Cost

The purpose of the proposed project is to carry out such an evaluation, using the best expertise that is available, in an exercise that will include comparisons both between different methods and different laboratories, with the aim of making 'best practice' recommendations.

Participants

A broad consortium will be formed with representation from four major groupings.

1. Companies that are suppliers of hardmetal powders (powder suppliers)
2. Companies using powders in the production of hardmetal tools (powder users)
3. Companies that market instruments for powder characterisation (instrument suppliers)
4. Research institutes and university departments having relevant expertise (research centres)

It is anticipated that there will be about 4 to 6 participating organisations in each of these groupings.

There will be, in addition, an independent coordinator for the programme.

Methodology

Batches of test material will be provided to the coordinator by the powder suppliers after consultation with the powder users. These powders should have average particle sizes targetted in the range 0.1 to 0.5 μm , although values outside this range can be included if there is a specific reason for doing so. On the basis that each supplier contributes two types of powder, the number of different materials in the investigation would be approximately 10 or 12.

Representative samples of the powders will be distributed to all the centres where measurements will be carried out on the following basis:

- Powder suppliers and powder users will use such of their available techniques as they consider most suitable.
- Instrument suppliers will apply the techniques that are most appropriate from their range of commercially available products.
- Research centres will be selected according to proven expertise in specific techniques.

All measurement techniques will be tested at a minimum of two organisations, There is no upper limit to the number of organisations applying any given technique

All results will be collated by the coordinator and reviewed critically.

The results and conclusions will be presented in a final report that will be distributed to all participating organisations.

Funding, organisation and time-scale

The present proposal is based on a previous review 'Characterisation of particle and grain size in the range nanometre to micrometre: a survey for the hardmetal industry' by B.Hutchinson and O.Grinder. That previous work was funded by a consortium of companies. It is intended that the present proposal will be open to other members of EPMA in addition to the original consortium. However, the contribution of the original consortium members will be recognised by a financial discount in the new project.

Each of the powder suppliers and powder users will pay a monetary contribution to the project via EPMA.

Any measurements carried out by powder suppliers and users will be a further contribution in-kind.

Measurements made by the instrument suppliers will be in-kind contributions to the project.

Research centres and the coordinator will receive payment for the work that they contribute to the project according to a schedule to be agreed.

Work will commence as soon as agreement has been reached and all participants have committed themselves to the project. A period of 9 months is allocated for completion of all experiments. The final report will then be completed and distributed within 3 months.