

Report

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**WC Powder Characterisation:
Technique Intercomparison**

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ABSTRACT

The manufacturing technology of hardmetal products is driven by tailoring their properties to performance requirements primarily through control of the WC grain size and Co content. There is increasing interest in hardmetal components with ever finer structures, especially sub 0.5 μm in WC diameter. Conventional measurement techniques, such as Fisher Sub Sieve Size, for characterising powders in this size range are working at their limit, if not below. Consequently, it is imperative that good practice is used for this and other complementary methods based, for example, on laser or surface gas adsorption (BET) techniques. An interlaboratory exercise has been conducted with participation by three global stakeholders: Sandvik Hard Materials, UK; Wolfram Bergbau- und Hütten GmbH, Austria; and Osram Sylvania, USA (now Global Tungsten Powders) in order to compare state-of-art powder characterisation techniques.

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Approved on behalf of the Managing Director, NPL,
by Dr M Cain, Knowledge Leader, Materials Team
authorised by Director, Operations

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1 INTRODUCTION

The objective of this study was to investigate method uncertainties to underpin good practice in the measurement of the size of WC powders in the range 0.2-0.5 μm . The activity had three core partners supported by NPL as coordinator on behalf of the European Hard Materials Group (EHMG) of the European Powder Metrology Association (EPMA). These three companies are

Wolfram Bergbau- und Hütten GmbH (WBH), Austria
 Sandvik Hard Materials, UK
 Osram Sylvania, USA (Global Tungsten Powders – GTP – as of Summer 2008)

A powder from a Japanese company (ALMT Corp) was also examined by microscopy for comparison.

The study was planned to proceed with the following steps:

- a WC powder manufacturers to each supply three powders in the size range 0.2-0.5 μm , together with measurement data.
- b NPL to collect 10 kg batches of each powder and circulate 1 kg of each to the three participating manufacturers for measurement of the powder characteristics. Data to be returned to NPL for central dissemination.
- c NPL to provide FEGSEM images of powders and conduct preliminary oxidation studies to assess the ability of this technique to characterise powders. Powders were also mounted in silver, sectioned and polished and imaged by scanning electron microscopy.
- d Results to be compared, discussed and method uncertainties quantified.
- e Good Practice to be distilled from outcomes of research.

The nine powders were separately coded from each manufacturer as follows:

WBH	SHM	GTP (Osram Sylvania)
CRC030-40	SHM008	SO4C
CRC025	SHM004	SO7V
CRC015	SHM002	BCO4

Details of the as-supplied powders are given in Appendix A. A summary of key data from the WBH and SHM powders as-supplied is given in Table 1.

Table 1 – Basic powder data as supplied by manufacturer

	WBH			SHM		
	crc030-40	crc025	crc015	shm008	shm004	shm002
BET specific surface area (m ² /g)	1.31	1.70	2.41	1.20	1.90	2.40
BET diameter, micrometre	0.29	0.23	0.16	0.32	0.20	0.16
FSSS number (µm) as supplied	0.80	0.65	0.51	0.95	0.75	0.56
FSSS (µm) ASTM deagglomerated	0.82	0.69	0.57	0.84	0.66	0.55
Pressing density (g/cm ³)	7.87	7.47	6.99			
Porosity (as supplied)	0.63	0.665	0.693	0.72	0.74	0.78
Porosity (ASTM deagglomerated)	0.6	0.623	0.663			
Free carbon (%)	0.03	0.04	0.04	0.03	0.03	0.01
Total carbon (%)	6.13	6.12	6.08	6.10	6.08	6.07

The GTP (Osram Sylvania) powders were coded as follows:

S04CV0790 0.70 µm FSSS plus 0.4 wt% VC_{0.88} / 0.5 wt% Cr₃C₂
S07V2408 0.90 µm FSSS plus 0.3 wt% VC_{0.88}
BC04U7012 0.63 µm FSSS (CILAS – 0.4 µm)

As-Supplied Information

Summary measurements from a reference published by GTP (Osram Sylvania) [1] have been replotted in Fig 1 to put the current exercise into context. BET and FSSS are gas adsorption and sieve methods respectively. X100 and UPA were laser diffraction and Brownian motion detection systems respectively.

It can be seen that the BET values were much smaller than those obtained from the optical and FSSS methods. The FSSS and BET methods were able to discriminate between the powders studied whereas the standard deviations associated with the optical methods [1] were too large to confidently separate the different powders. A similar comparative plot is shown in Fig 2 for the as-supplied data from WBH and SHM. It shows a similar pattern and discrimination to that observed by Osram Sylvania [1]. The BET measurements of grain size were much smaller than those obtained from the optical and FSSS methods.

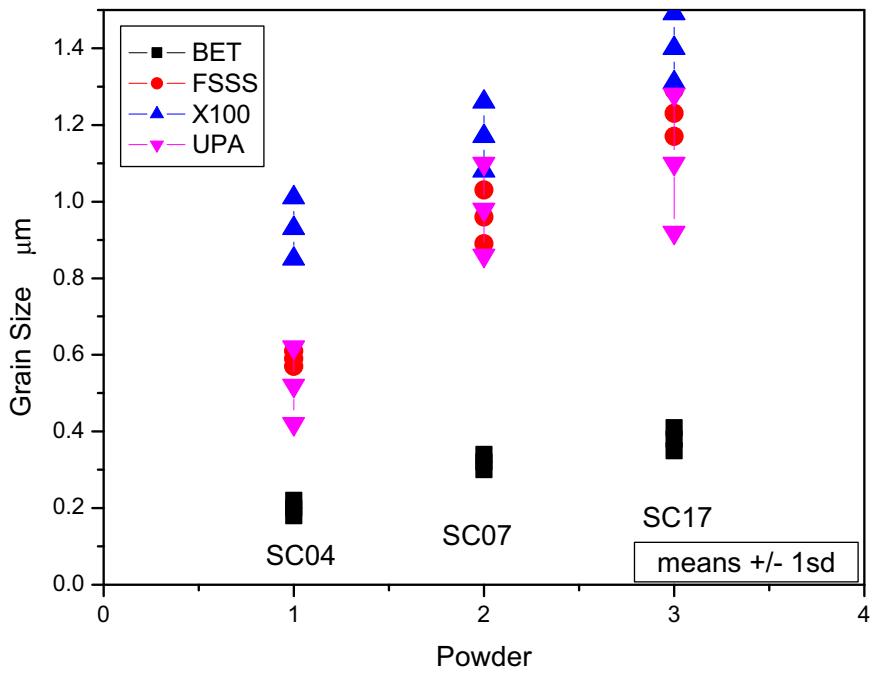


Fig 1 Comparative data from GTP (Osram Sylvania) [1], standard deviation indicated by vertical lines.

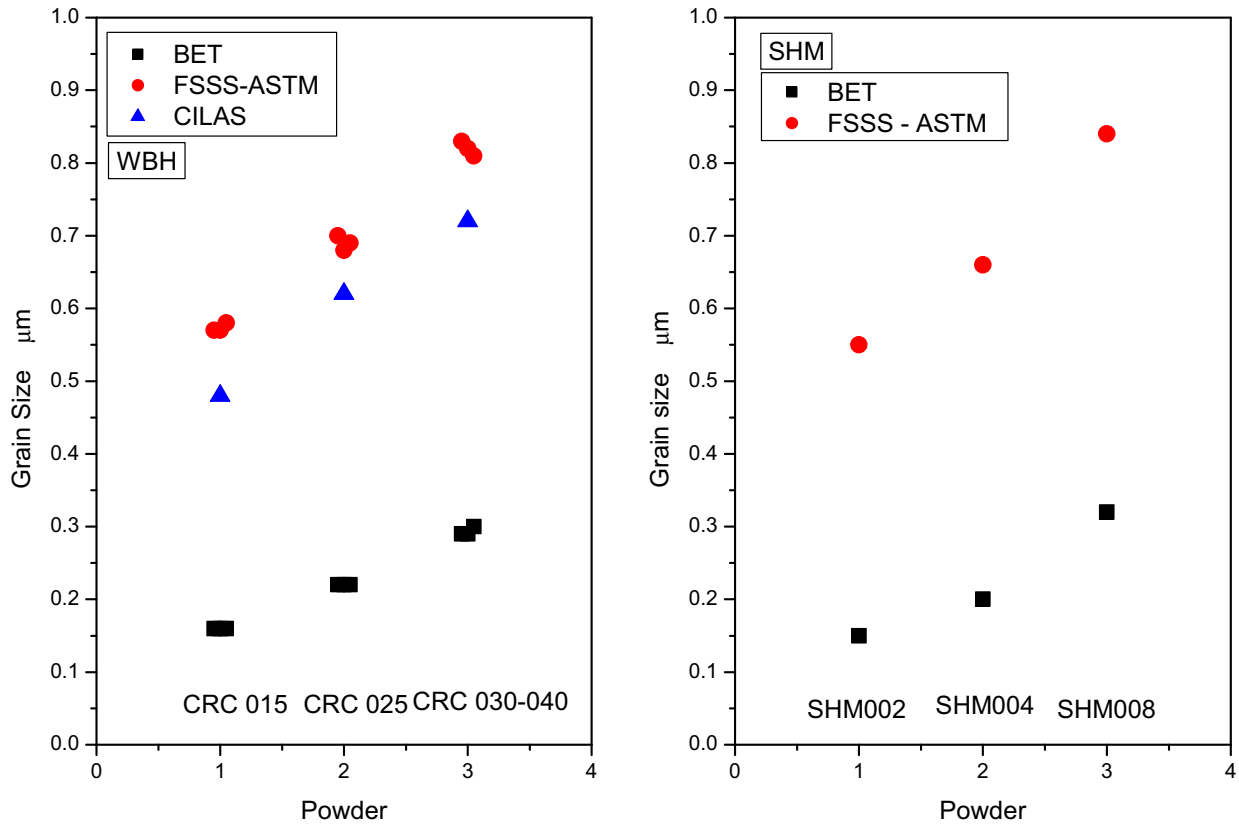


Fig 2 Comparative measurements from WBH and SHM

2 NPL OXIDATION STUDIES

Previous work at NPL has shown that the oxidation kinetics of WC powders can be related to grain size and size distribution [2-4]. However, these studies had been conducted in a thermogravimetric analyser (TGA). TGA equipment is rather specialised and not widely available. Consequently for the current exercise a simpler approach was explored in which 30 mg (nominal) samples of each powder were placed in a platinum boat and then exposed to air in a laboratory furnace at 450 °C. The change in mass as a function of time was recorded (Figs 3-5). Also included in Figs 3-5 is data from a Japanese sourced powder (ALMT Corp). The ALMT powder had a BET grain size of 0.1 micrometres and images are also shown in Appendix B.

***Note:** repeat measurements indicated that the mass changes were quite sensitive to the precise position of the platinum boat in the furnace. The furnace was quite large in size and the platinum boat sat on a ceramic plate within the furnace. Its position can be a few centimetres different from run to run. At 450 °C there are likely to be substantial temperature gradients within the furnace heated zone. Further studies are needed, perhaps to use a smaller tube furnace, to examine repeatability issues.*

A nominal correlation was obtained between the time to 50% mass change and the BET particle size measurements reported later, but further work in a more controlled furnace space would be needed to confirm this observation.

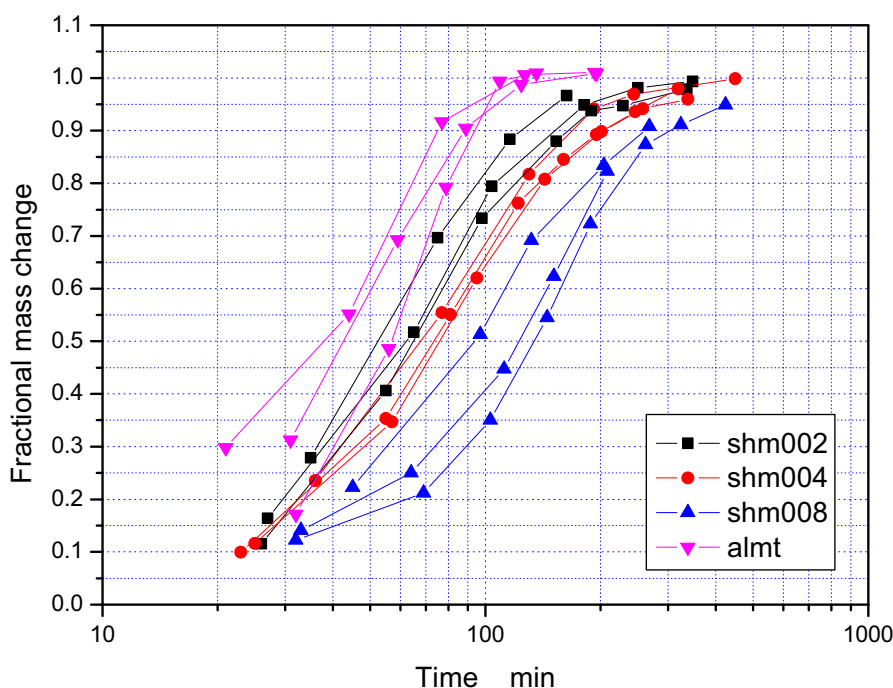


Fig 3 Example of change in mass plotted against time in oxidation experiments on SHM powders at 450 °C.

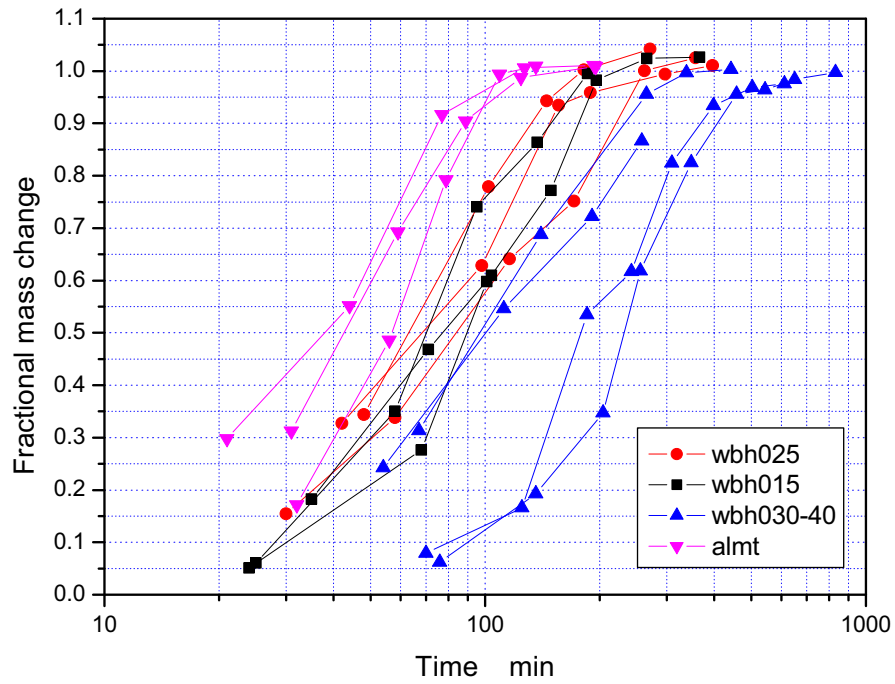


Fig 4 Example of change in mass plotted against time in oxidation experiments on WBH powders at 450 °C.

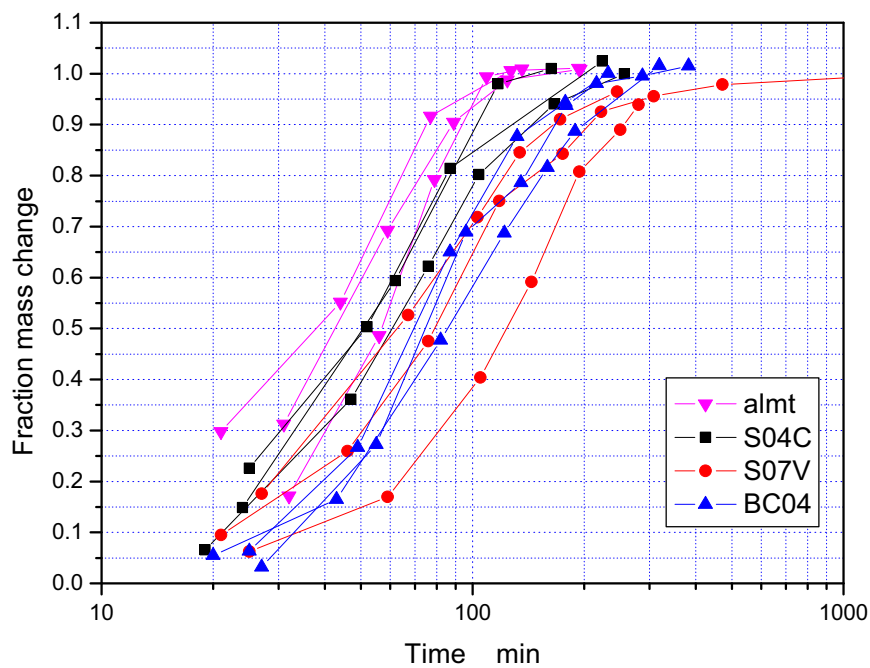


Fig 5 Example of change in mass plotted against time in oxidation experiments on GTP (Osram Sylvania) powders at 450 °C.

Because of the uncertainties associated with positioning the small platinum container within a large furnace it was decided to repeat the oxidation measurements in a TGA system whereby temperature was ramped at 0.1 °C/s in flowing air and the mass change was monitored till completed oxidation had occurred by about 700 °C. Plots of relative mass change for three repeatability experiments on one powder grade from each of the powder suppliers is shown in Fig 6 indicating good repeatability. It can be seen that the SHM002 powder has a slower approach to the final mass value implying that it contains some larger agglomerates or particles that take longer time to oxidise.

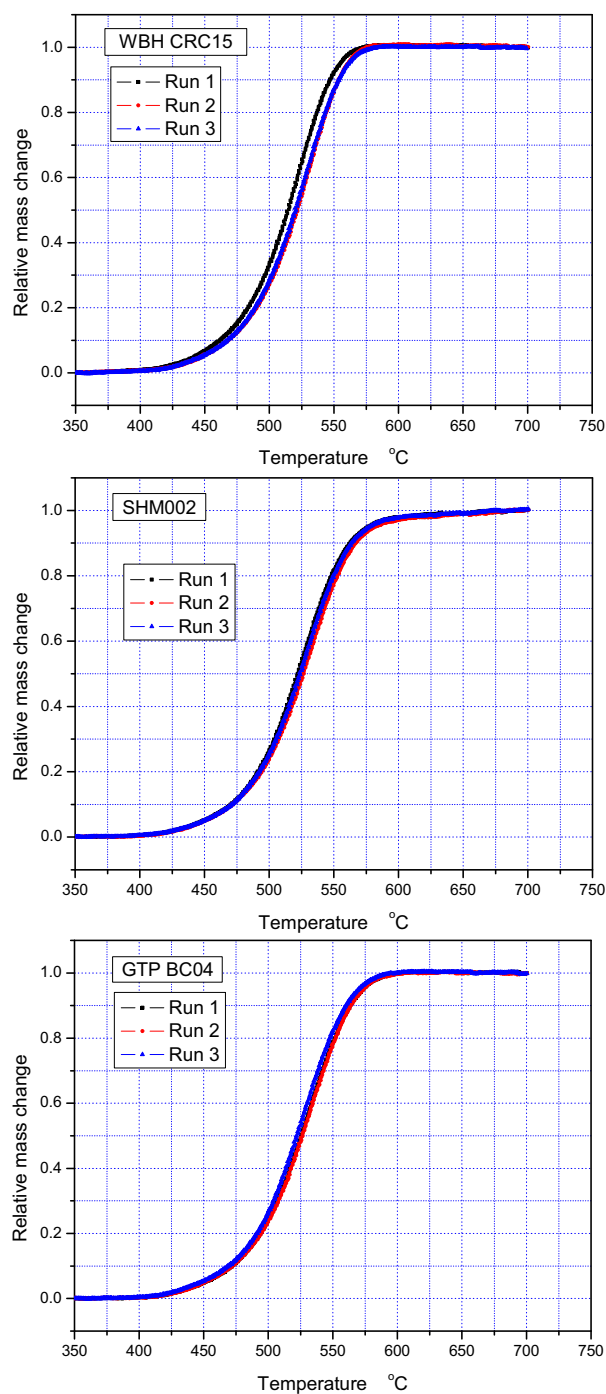


Fig 6 Relative mass change in three repeatability ramped TGA experiments on one powder grade from each powder supplier.

Comparative results for the nine powders grouped by organisation are shown in Fig 7. For the SHM and WBH powders there is a systematic increase in rate of oxidation as the nominal size of the powder decreases. However, for the GTP powders, GTP BC04 stands out as being narrower in size distribution (steeper curve) and is out-of-line with respect to the nominal sizes of the three GTP powders.

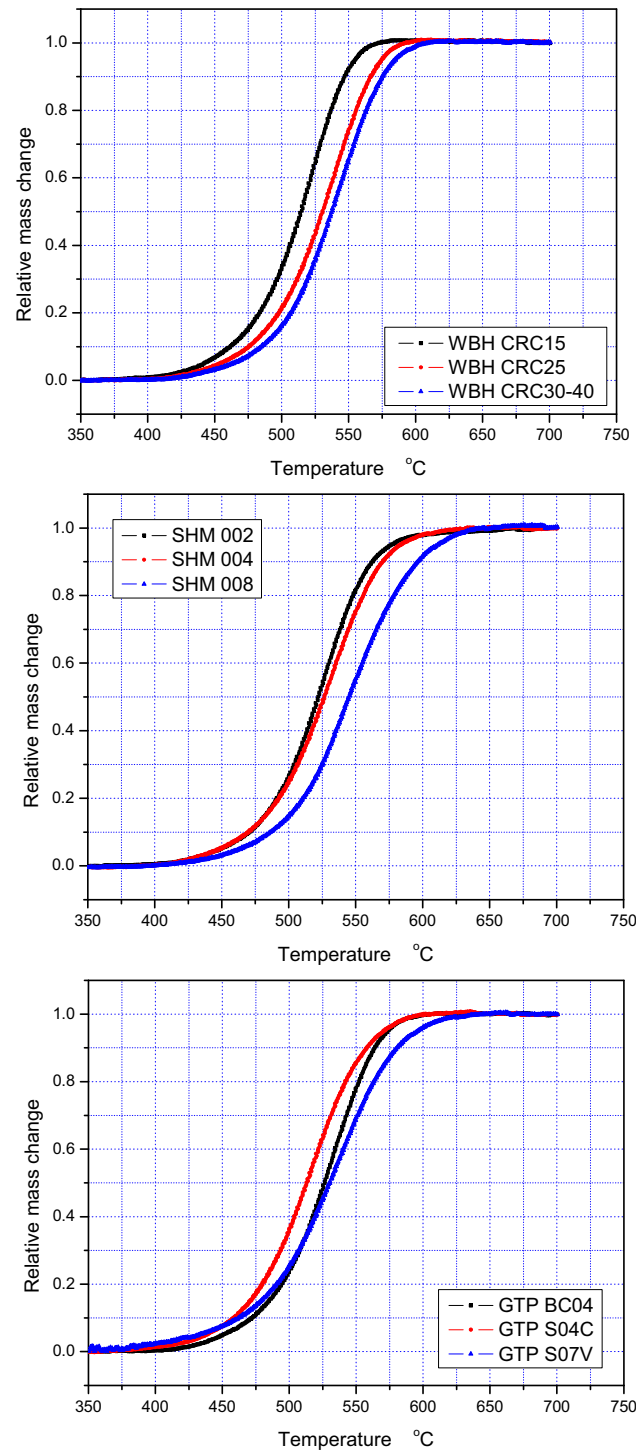


Fig 7 Relative mass change in ramped TGA experiments on all powders, grouped by organisation.

Comparative results for the nine powders grouped by nominal size are shown in Fig 8. The WBH powders appear to have a narrower size distribution (steeper oxidation plots) than the SHM and GTP powders.

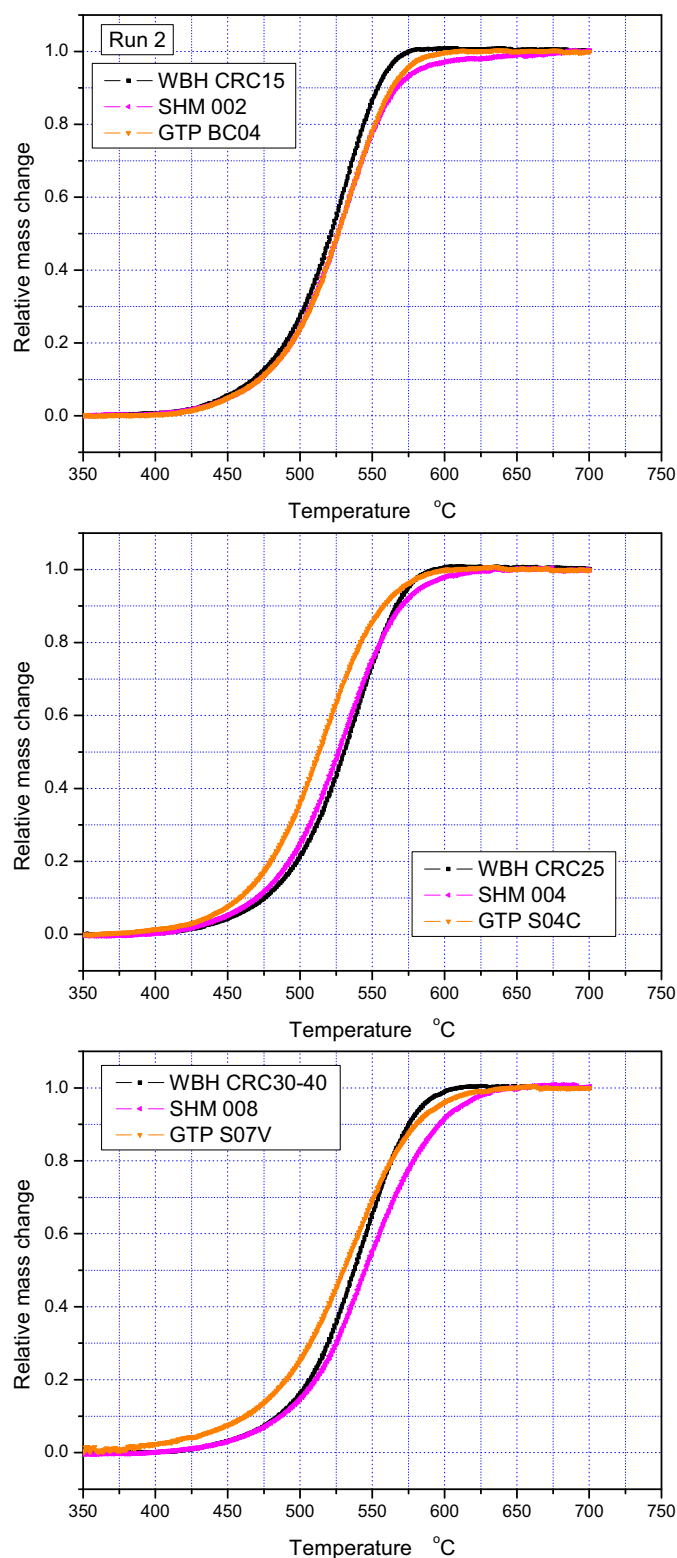


Fig 8 Relative mass change in ramped TGA experiments on all powders, grouped by nominal size (finest at top, coarsest at bottom).

In order to compare the kinetic data a plot was constructed (Fig 9) of the temperature ($T_{0.5}$) at which 50% of the relative mass had been converted to oxide against the BET grain sizes reported by the interlab partners (see Appendix D). The WBH and SHM data points fall on a similar trend line together with GTP BC04. Samples SO4C and SO7V have generally lower $T_{0.5}$ values, probably because these two powders contain additional cubic carbides and their presence slightly affects the oxidation kinetics.

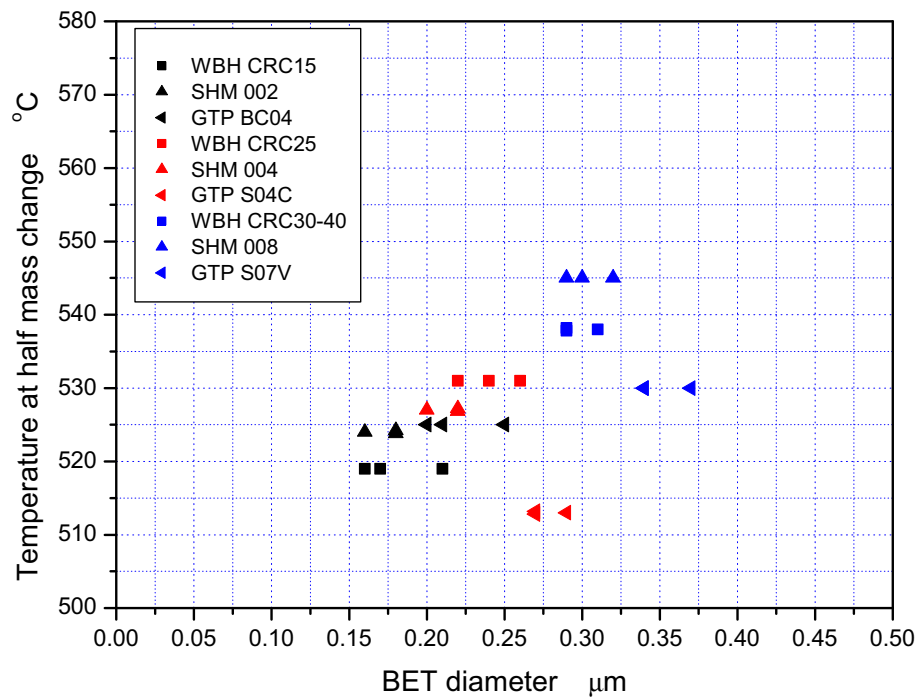


Fig 9 Temperature at which 50% mass conversion had occurred compared with BET grain size.

3 MICROSTRUCTURE

3.1 NPL FEGSEM IMAGES

Each of the powders was imaged using a high resolution scanning electron microscope (FEGSEM: Zeiss Supra) at a range of magnifications. The powders were simply shaken onto a metal plate/stub which was covered with a small amount of a conducting adhesive. A set of micrographs is attached as Appendix B.

3.2 NPL POWDER SIZE MEASUREMENTS ON POLISHED SECTIONS

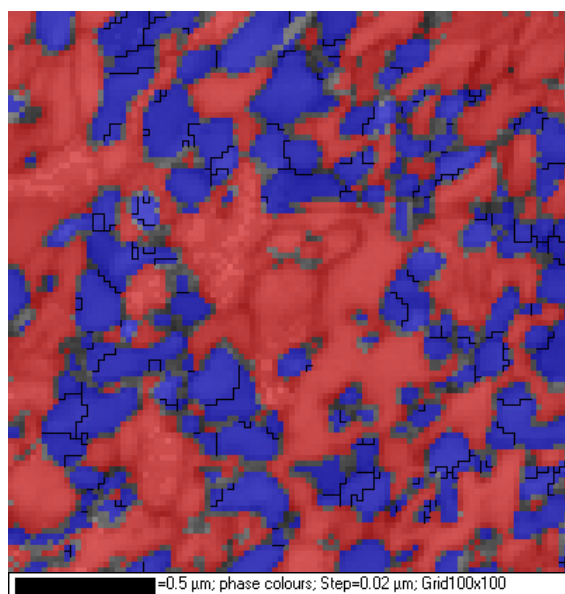
Samples of the powders were mounted in silver and sectioned and polished for imaging by FEGSEM and EBSD – Electron Backscatter Microscopy [5]. Figure 10 shows typical images of the ALMT powder with phase and EBSD orientation coded information. The EBSD maps were obtained with a very small step size of 20 nm. Figure 11 shows the size (equivalent circle diameter-ECD) distribution data obtained with an even smaller step size of 10 nm. The

data has been plotted both with all grains counted and with grains less than 5 pixels removed (arbitrarily chosen as noise or below resolution limits). However, it can be seen that theoretical sections through an equiaxed particle distribution (Fig 12) have a distinctive curvature similar to that shown in Fig 11. In the EBSD data $\Sigma 2$ boundaries (where there is a strict orientation relation across the boundary [6]) have been included as grain boundaries or not, as the case may be (Table 2). FEGSEM images of the sectioned WBH, SHM and GTP powders are shown in Appendix C. EBSD maps are shown in Appendix D. Grain size measurements were obtained from the EBSD images and the results are shown as number distributions, ignoring $\Sigma 2$ boundaries, in Fig 13. Mean values of grain size by equivalent circle diameter (ECD) are given in Table 2, with and without $\Sigma 2$ boundaries counted.

Table 2 – EBSD Particle Sizes; Equivalent Circle Diameter (ECD)

	$\Sigma 2$ boundaries counted			$\Sigma 2$ boundaries ignored			% grains without $\Sigma 2$
	Mean (μm)	Max (μm)	Number	Mean (μm)	Max (μm)	Number	
ALMT	0.076	0.30	304	0.089	0.30	224	74
CRC15U	0.14	0.51	518	0.18	0.65	327	63
CRC25U	0.16	0.73	2299	0.19	1.29	1490	65
CRC30-40	0.23	1.12	5093	0.27	1.39	3730	73
SHM002	0.13	0.54	499	0.15	0.64	382	77
SHM004	0.15	0.61	1092	0.18	0.62	786	72
SHM008	0.17	1.01	797	0.20	1.25	522	65
BC04	0.17	0.53	403	0.20	0.68	282	70
SO4C	0.15	0.67	1636	0.19	0.85	937	57
SO7V	0.23	1.04	8844	0.28	1.13	7132	81

Ag – red, WC – blue



WC Orientation Map

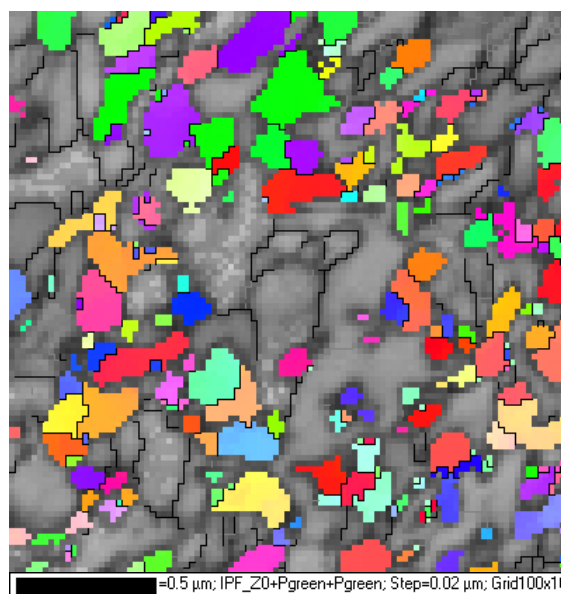


Fig 10 EBSD maps of sectioned ALMT powders mounted in silver.

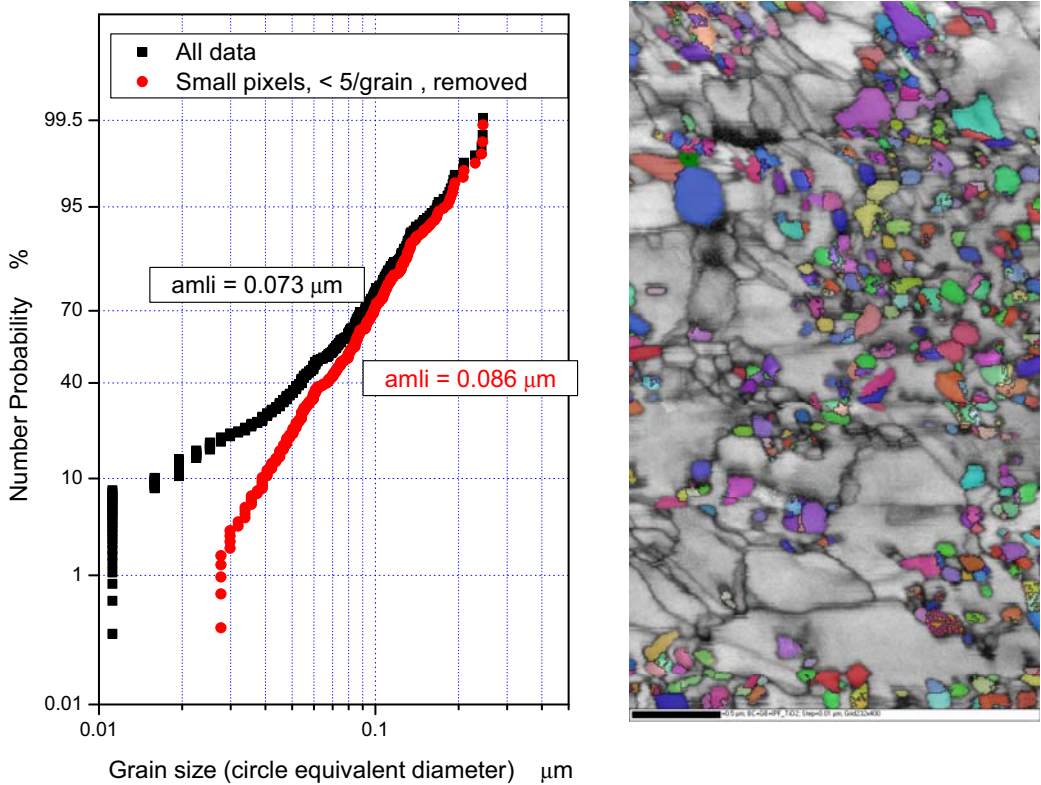


Fig 11 Size distribution data for sectioned ALMT powder and high resolution image of silver mounted powder.

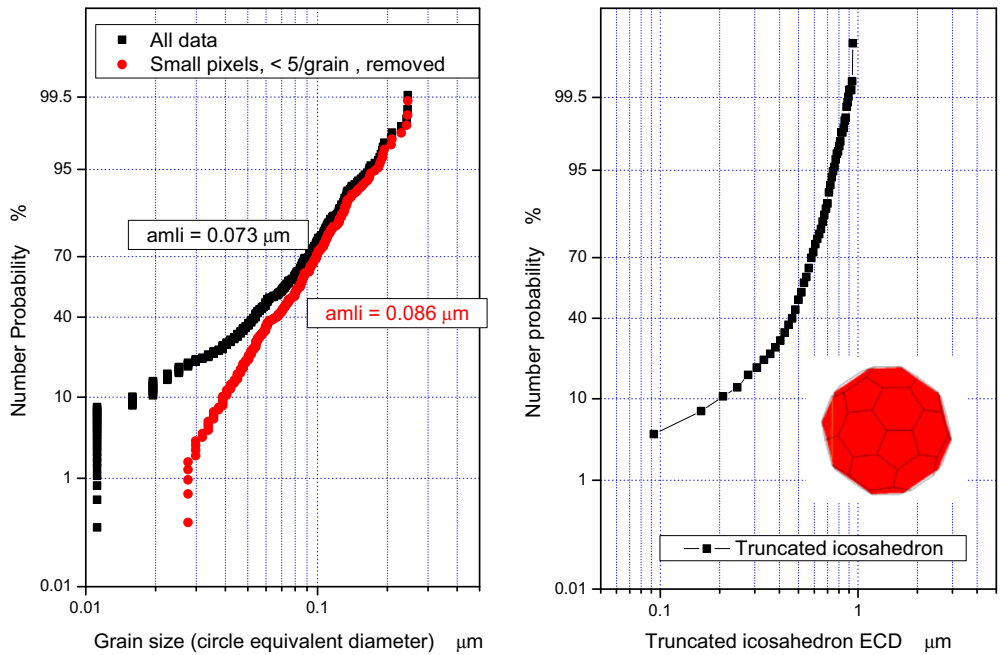


Fig 12 Comparison of measurements and model calculations.

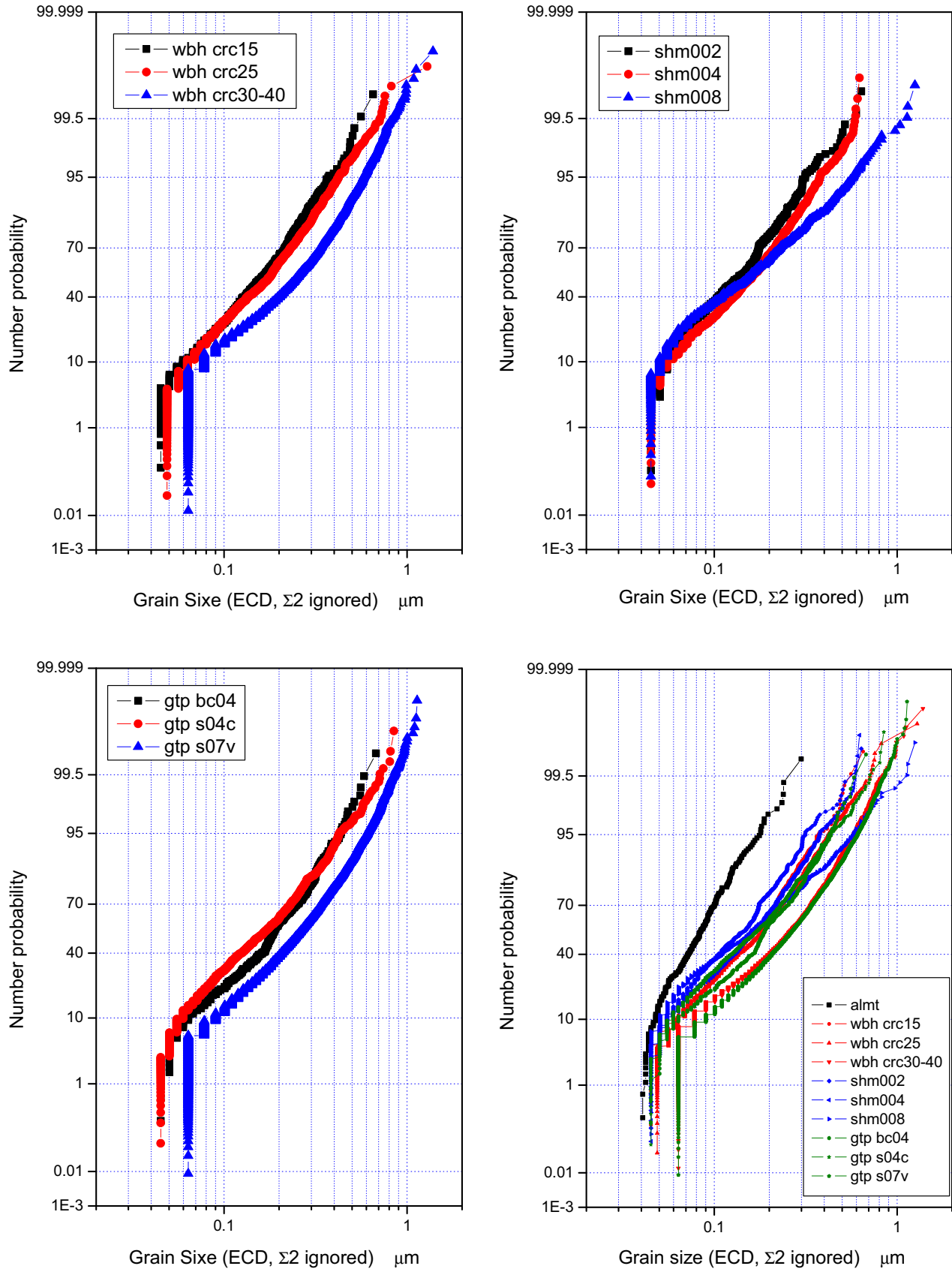


Fig 13 Number size distributions from sectioned powders.

4 INTERLABORATORY MEASUREMENT DATA

Results of the interlaboratory measurements supplied by partners WBH, SHM and GTP (formerly Osram Sylvania) are given in Appendix D. Comparative plots of information from these data sets are shown in Figs 14-20. The EBSD grain diameters (ECDs) have been multiplied by a factor of 1.33 to convert to a 3D value in Fig 20.

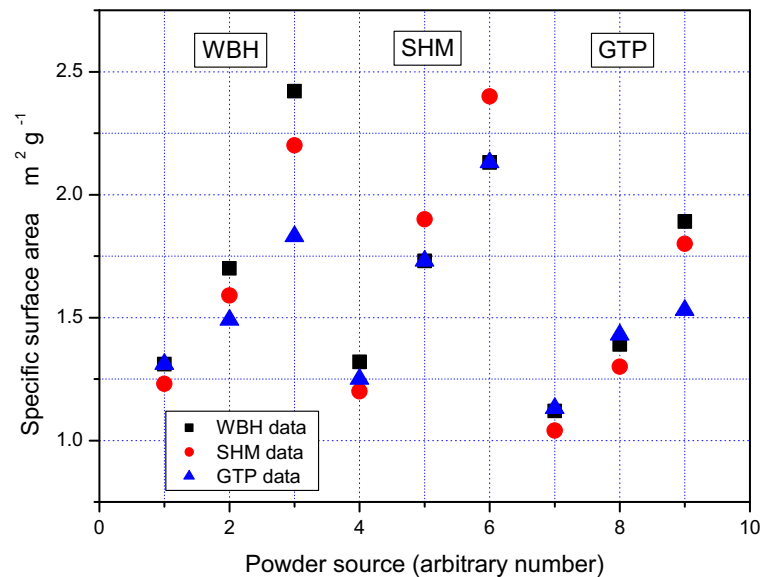


Fig 14 BET specific surface area (SSA).

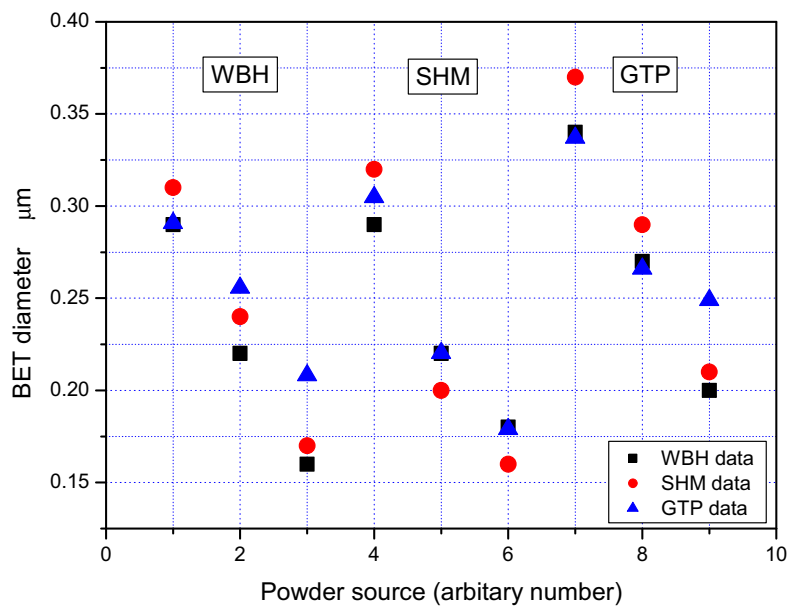


Fig 15 BET diameters (calculated from SSA).

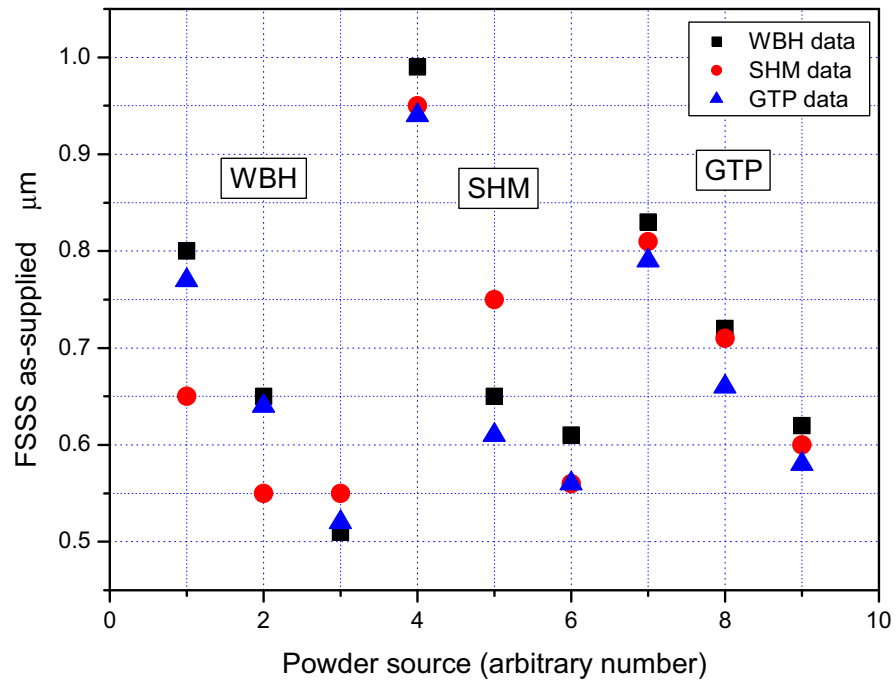


Fig 16 FSSS (as-supplied powders).

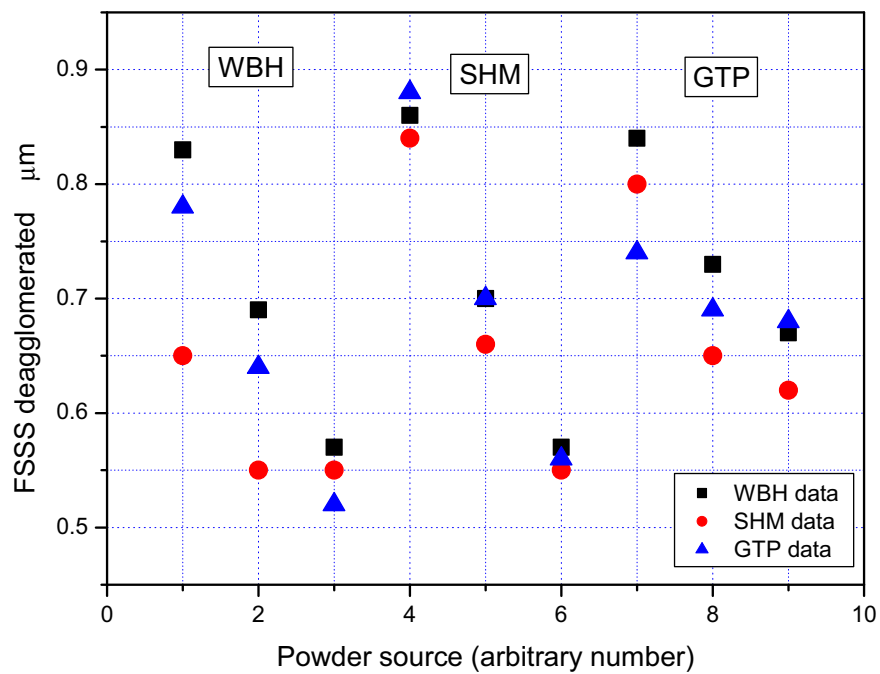


Fig 17 FSSS (de-agglomerated powders).

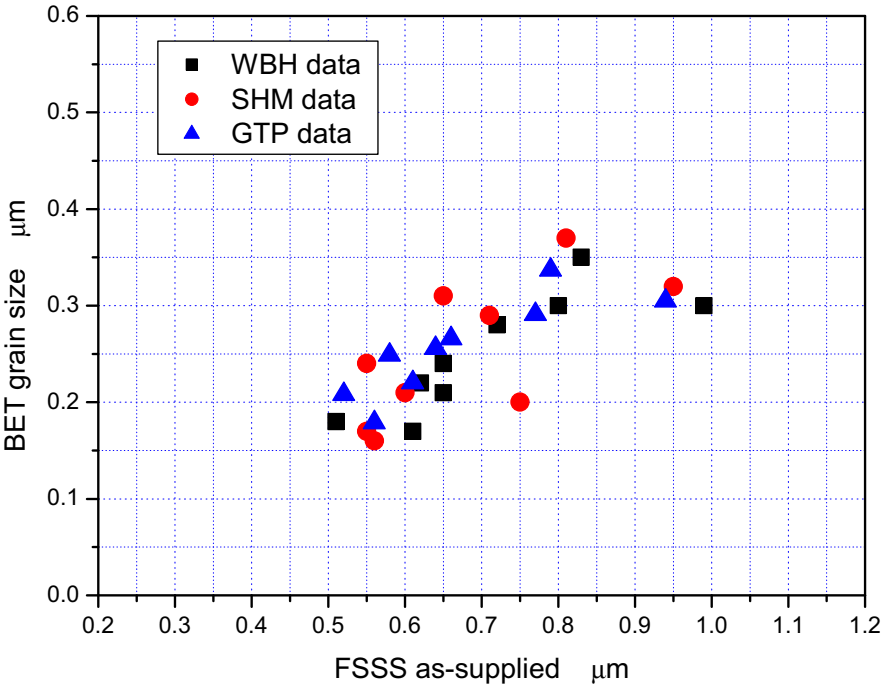


Fig 18 BET grain size (particle diameter) plotted against FSSS (as supplied).

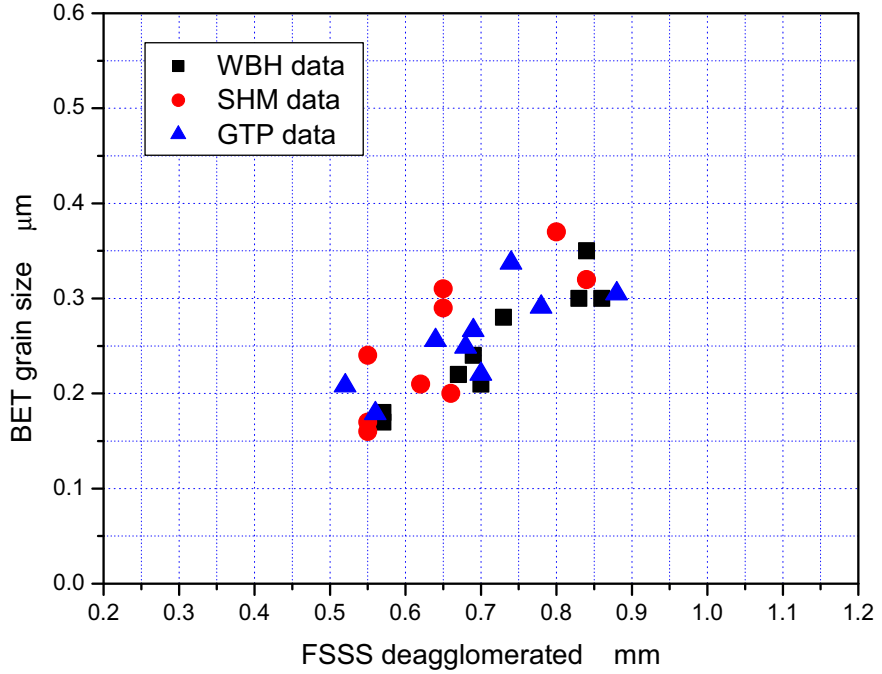


Fig 19 BET grain size (particle diameter) plotted against FSSS (de-agglomerated).

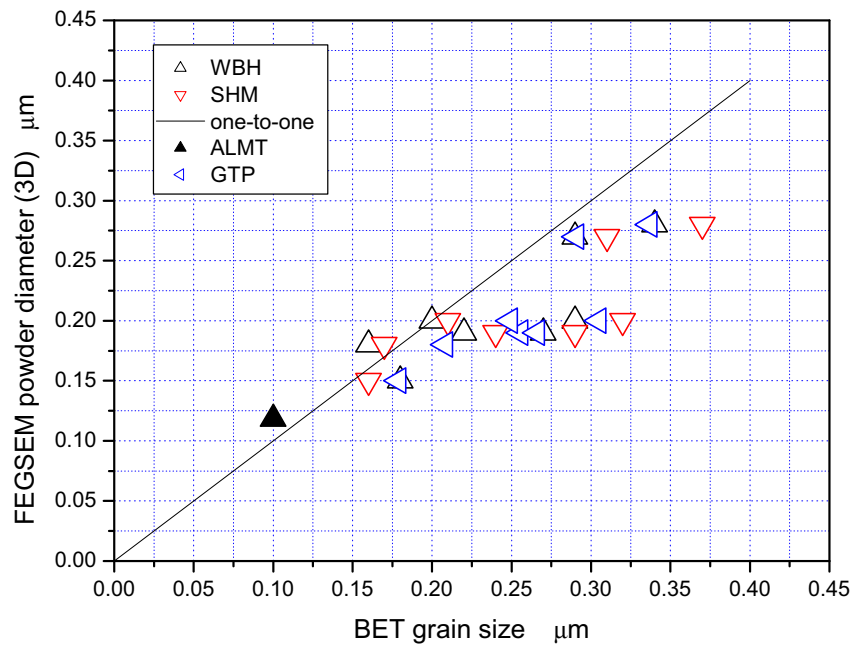


Fig 20 Comparison of EBSD grain sizes ($\Sigma 2$ boundaries ignored) and BET diameters.

Measurements of powder size distributions by laser diffraction were conducted by GTP (Osram Sylvania) and WBH. Figs 21-23 show the histograms for each set of powders (WBH, SHM and GTP respectively) from the GTP data, and only one plot for each pair of measurements has been shown, e.g. 7906 from 7906/7902 for CRC15, etc, while Figs 24-26 show cumulative distributions (where RM = rod milled) obtained from the Microtrac X100 system [1] and Figs 27-29 from the GTP data from the Microtrac S3500 system [1]. Fig 30-38 show typical plots for the laser diffraction data measured at WBH with the CILAS system. Figs 39-41 show comparative cumulative plots from the CILAS data for each organisation's powders. Clearly rod milling reduces the measured sizes slightly. Fig 42 compares the 50 wt% cumulative medians from each system (Appendix D) for every one of the powders. The following observations were made from the results shown in Figs 21-42.

- The laser systems had more difficulty measuring the SHM powders with multiple peaks detected, probably due to the presence of agglomerates.
- A typical standard deviation median particle size (between laser systems) is about $\pm 0.1 \mu\text{m}$ for the 50 wt% values, for particles in the range $0.4\text{-}0.8 \mu\text{m}$.
- Rod milling reduces the measured median value by about $0.05 \mu\text{m}$ and probably broke up some large agglomerates in SHM 004 powder.
- There are systematic differences between the different laser systems (Microtrac X100 > CILAS > Microtrac S3500).
- The cumulative plots show good repeatability for each powder, but different distributions were found, dependent on laser system used.

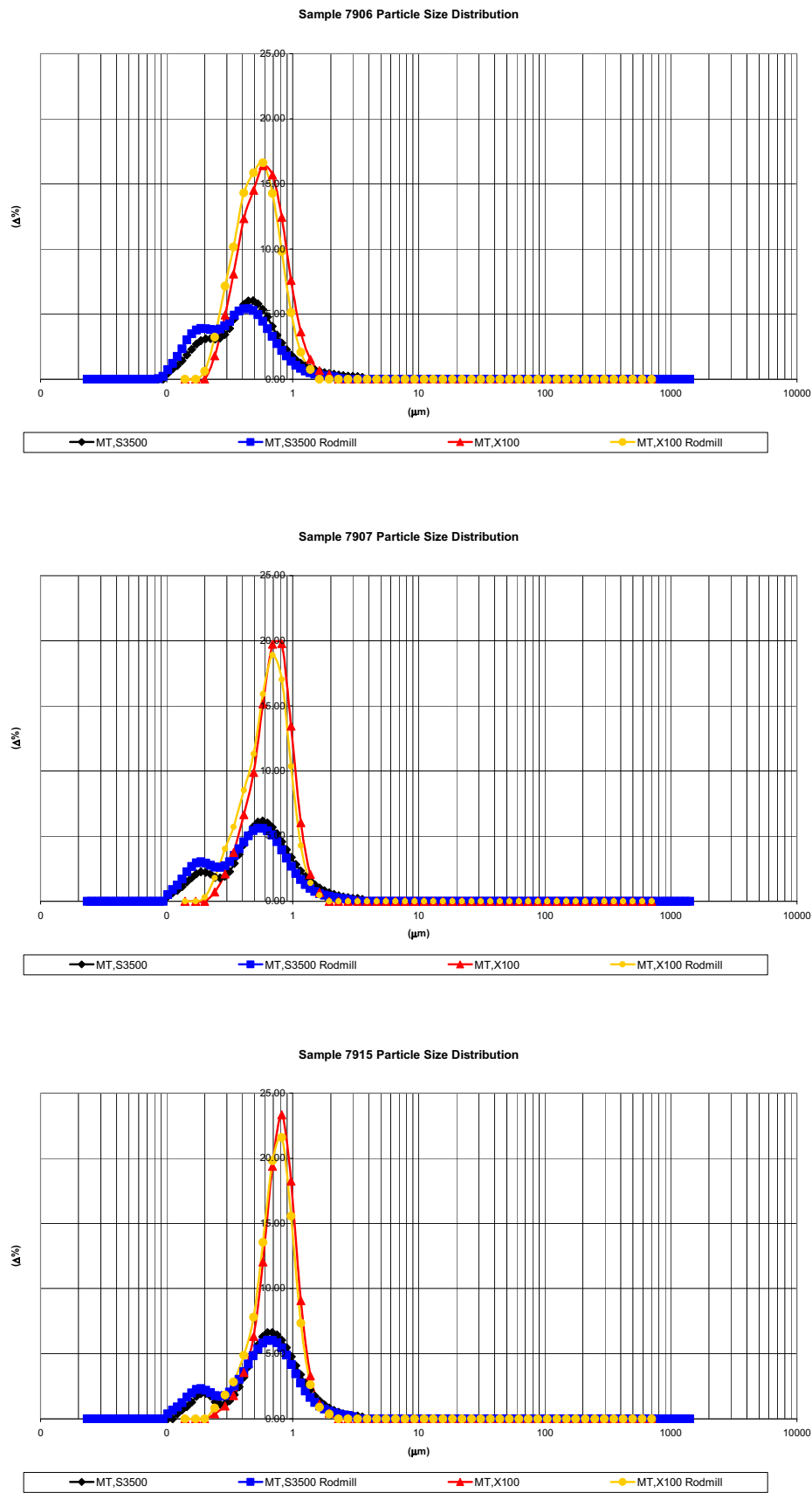


Fig 21 Laser diffraction data: Top (CRC15); Middle (CRC25); Bottom (CRC30-40).

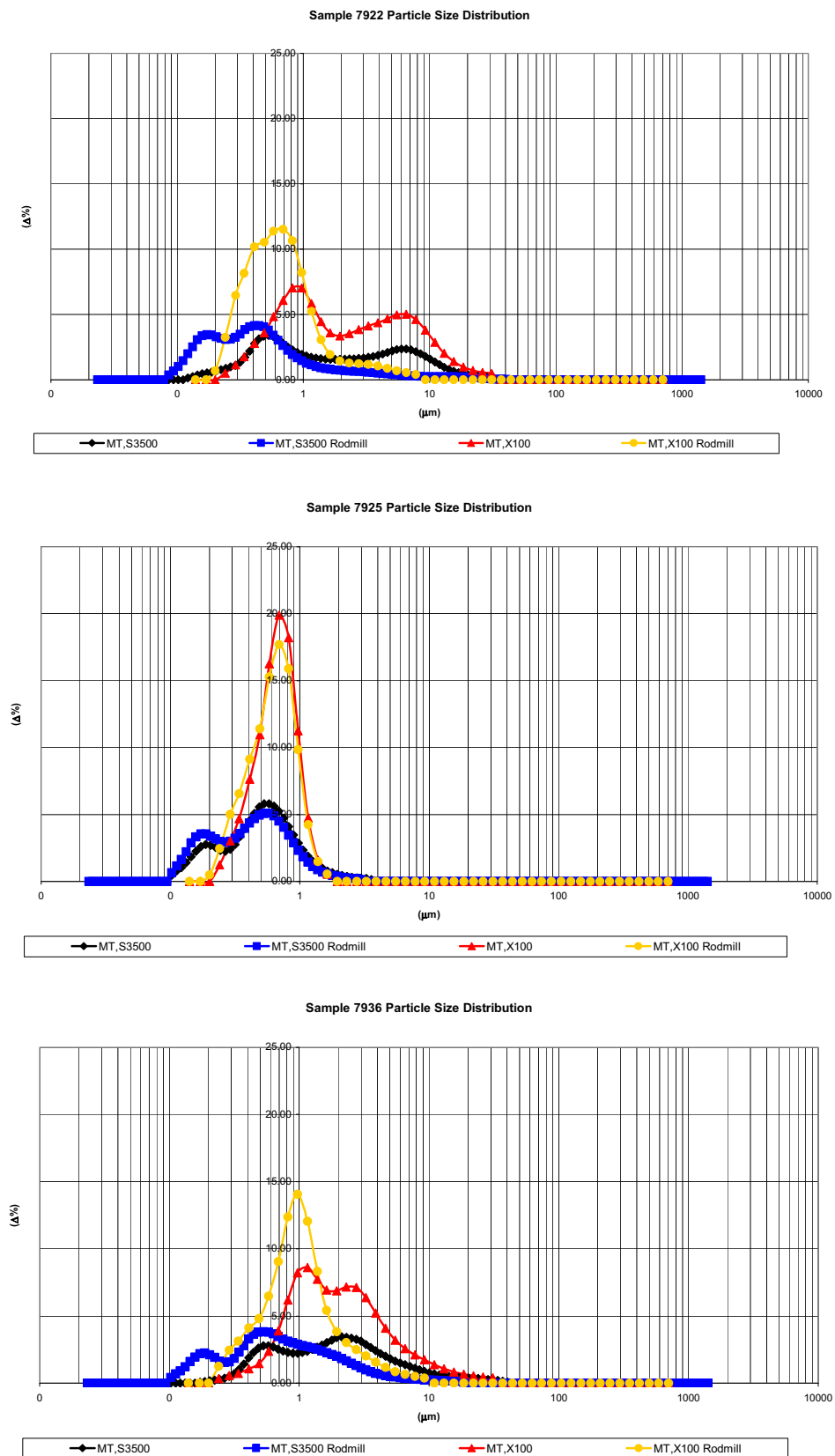


Fig 22 Laser diffraction data: Top (SHM002); Middle (SHM004); Bottom (SHM008).

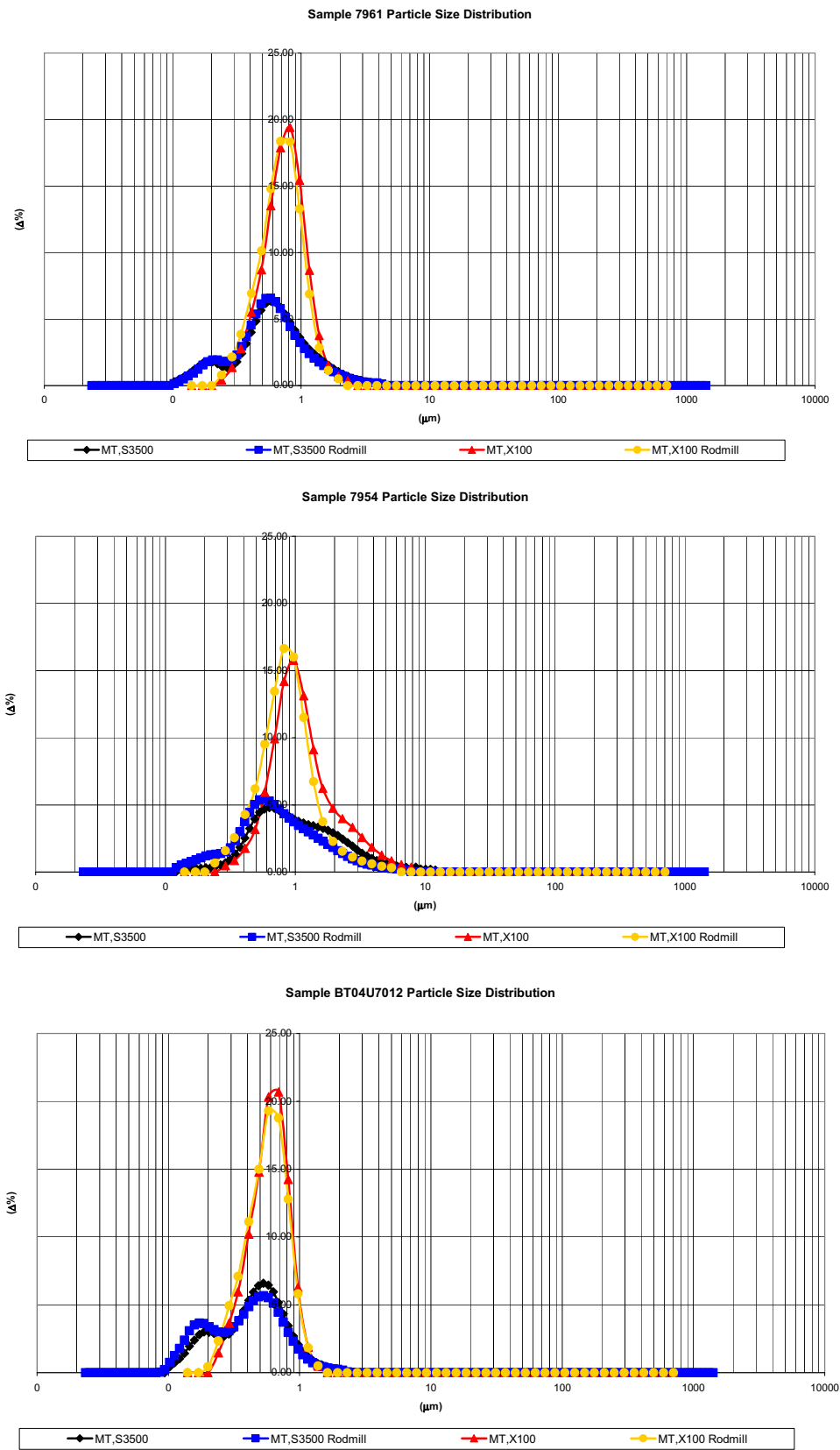


Fig 23 Laser diffraction data: Top (SO4C); Middle (SO7V); Bottom (BC04).

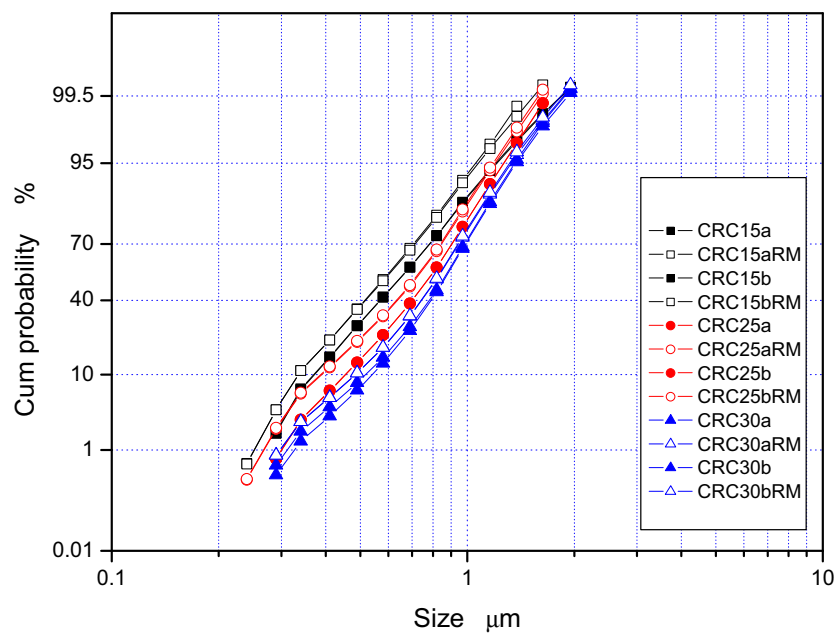


Fig 24 WBH powders: cumulative distributions; Microtrac X100.

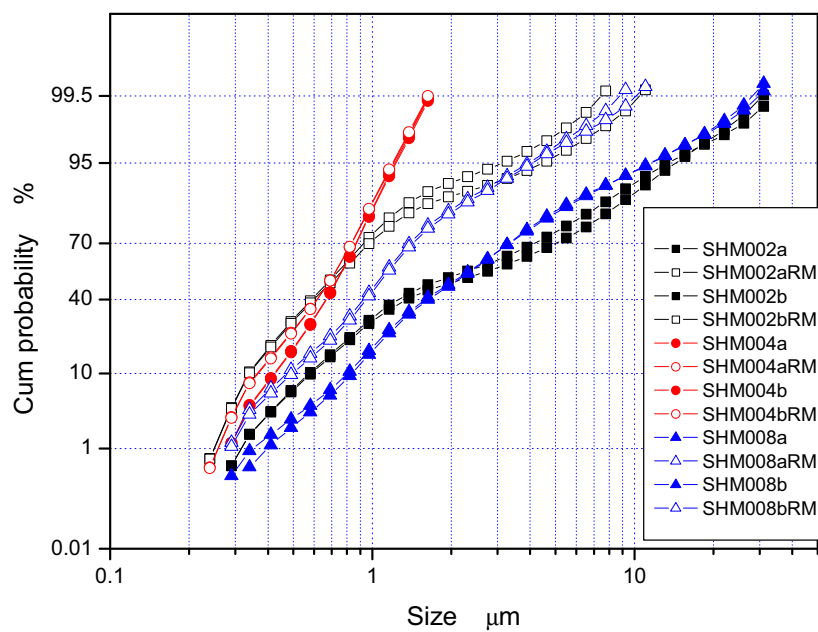


Fig 25 SHM powders: cumulative distributions; Microtrac X100.

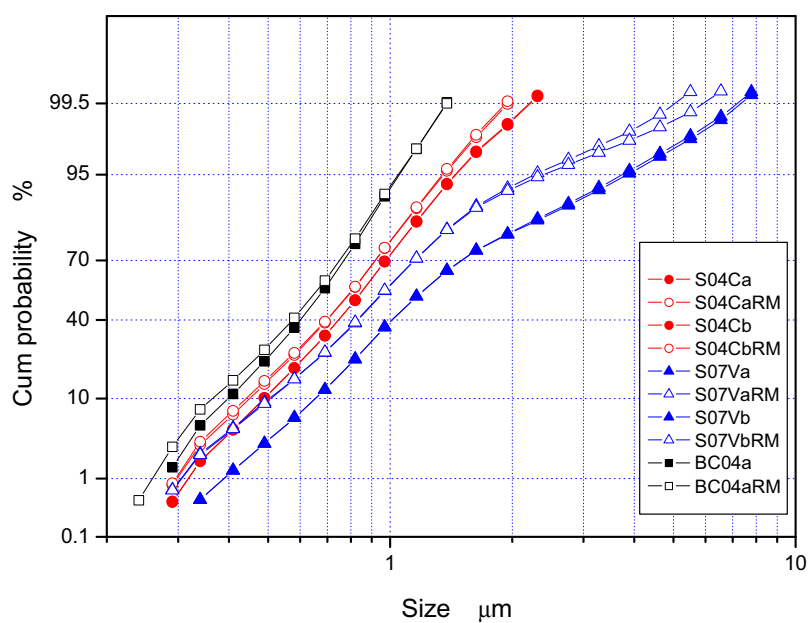


Fig 26 GTP powders: cumulative distributions; Microtrac X100.

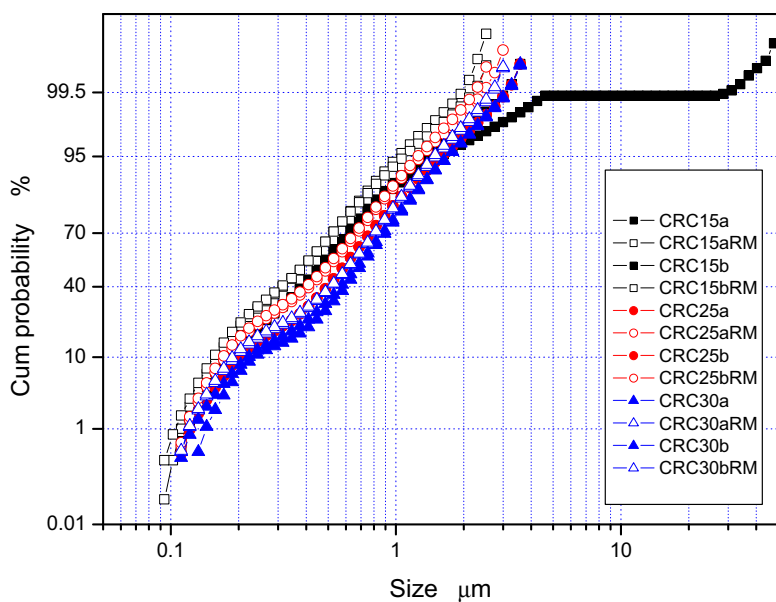


Fig 27 WBH powders: cumulative distributions; Microtrac S3500.

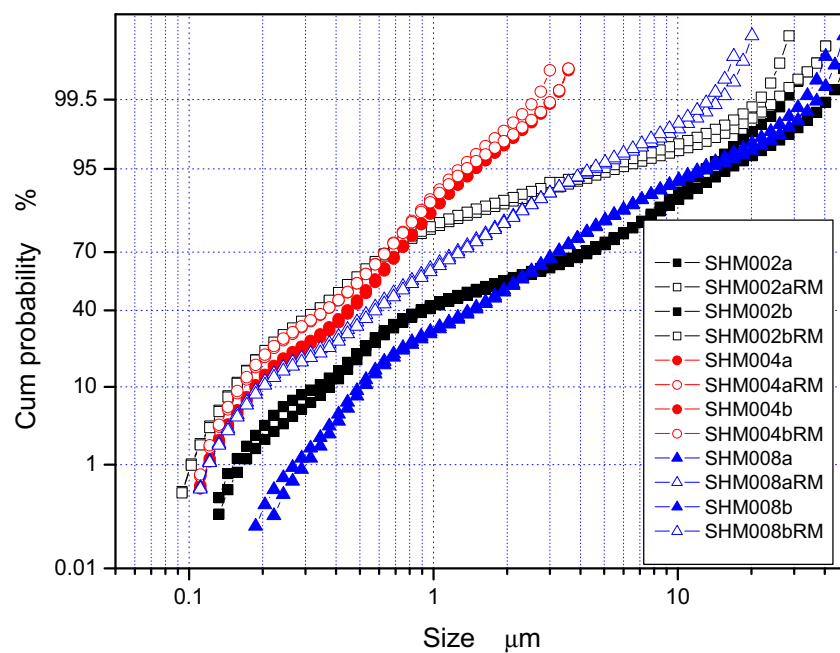


Fig 28 SHM powders: cumulative distributions; Microtrac S3500.

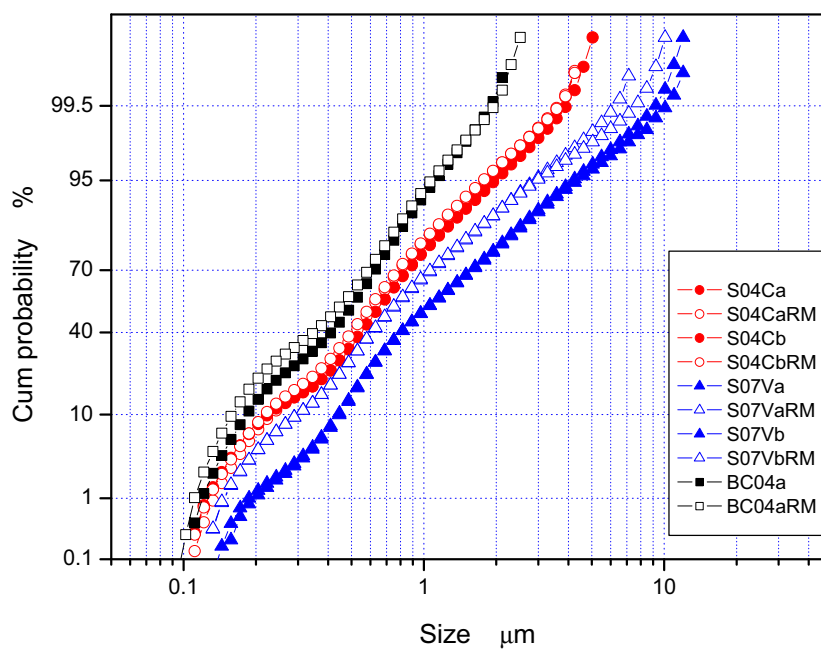


Fig 29 GTP powders: cumulative distributions; Microtrac S3500.

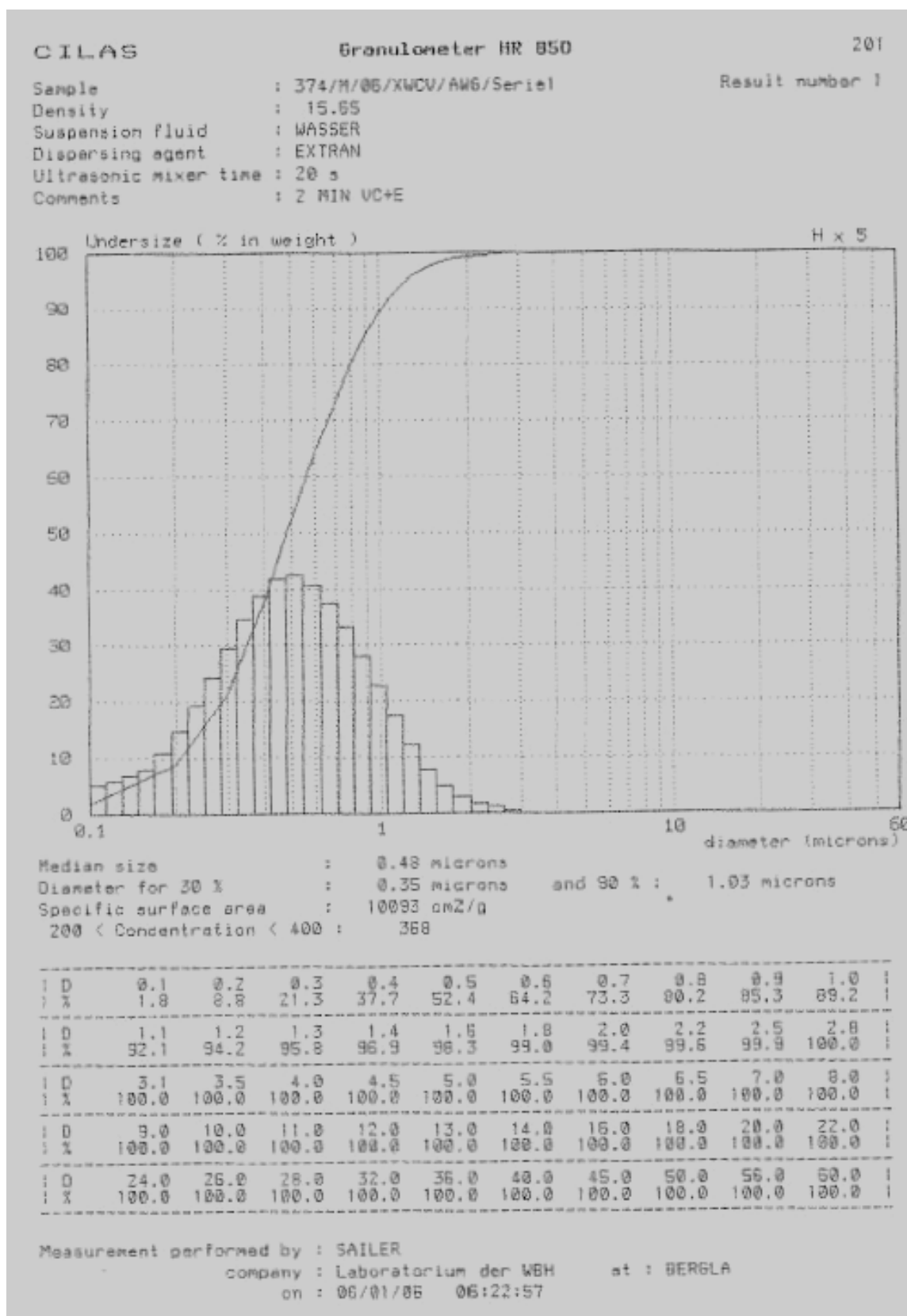


Fig 30 CILAS laser diffraction data for powder WBH CRC015.

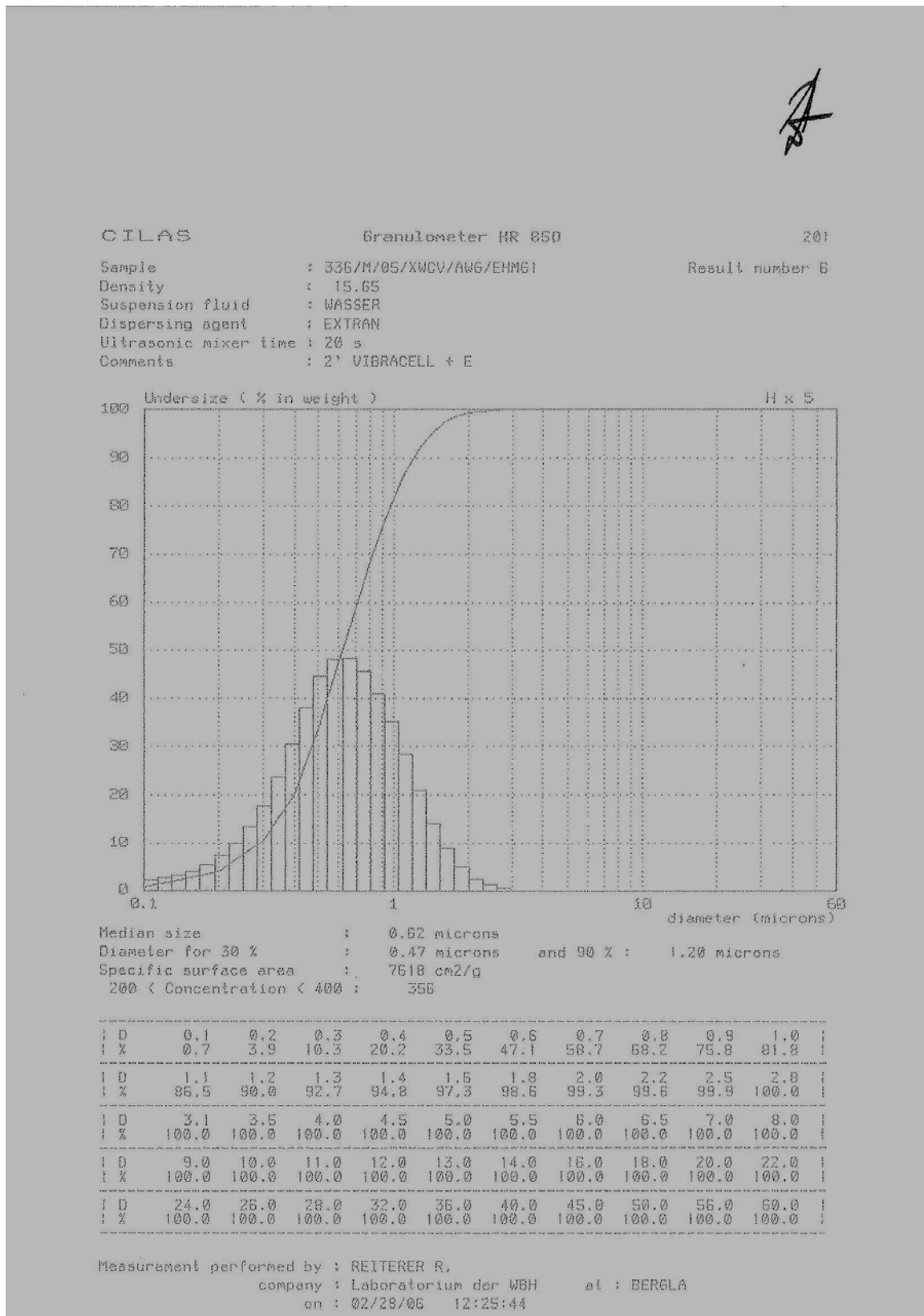


Fig 31 CILAS laser diffusion data for powder WBH CRC025.

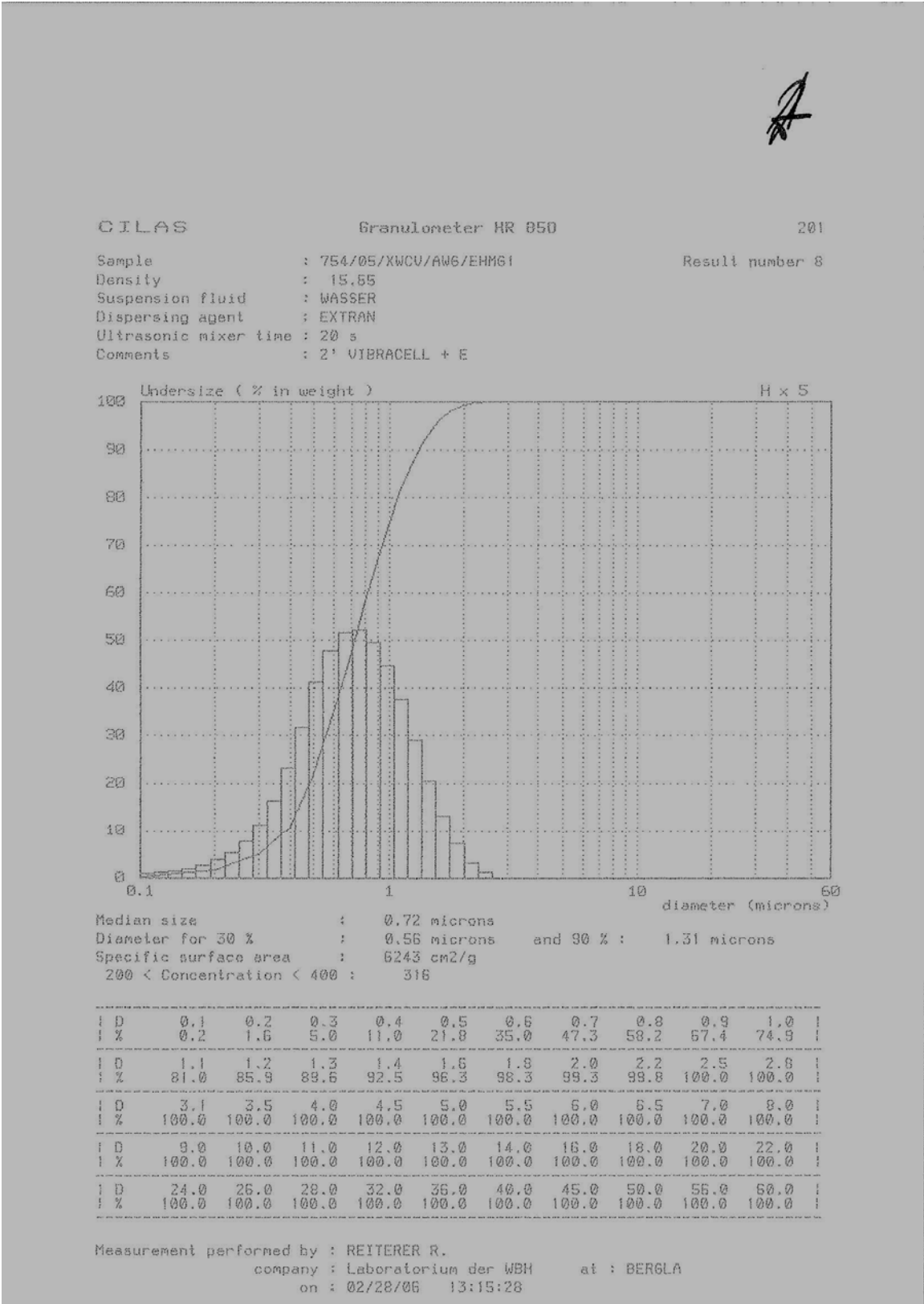


Fig 32 CILAS laser diffusion data for powder WBH CRC030-40.

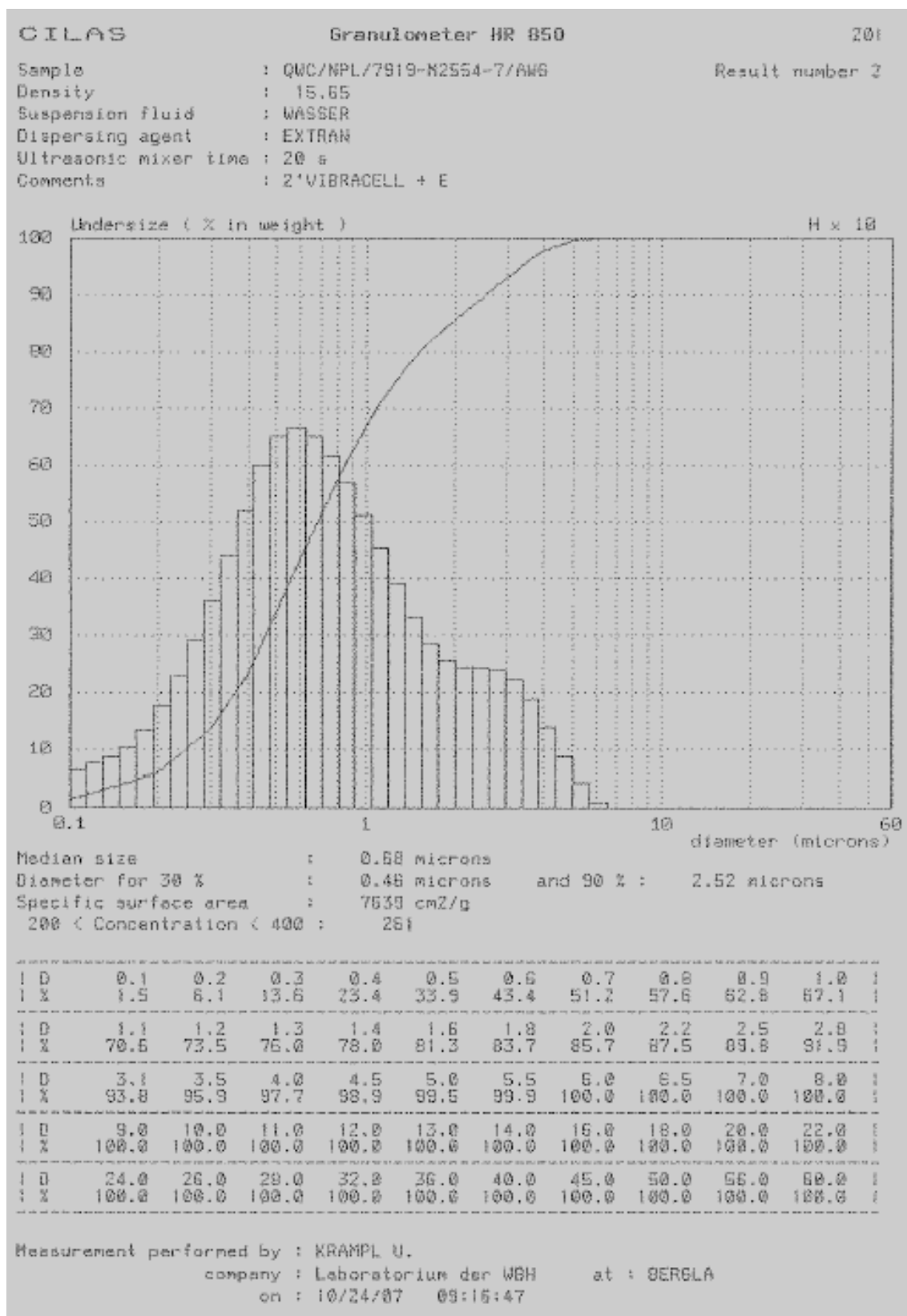


Fig 33 CILAS laser diffraction data for powder SHM 002.

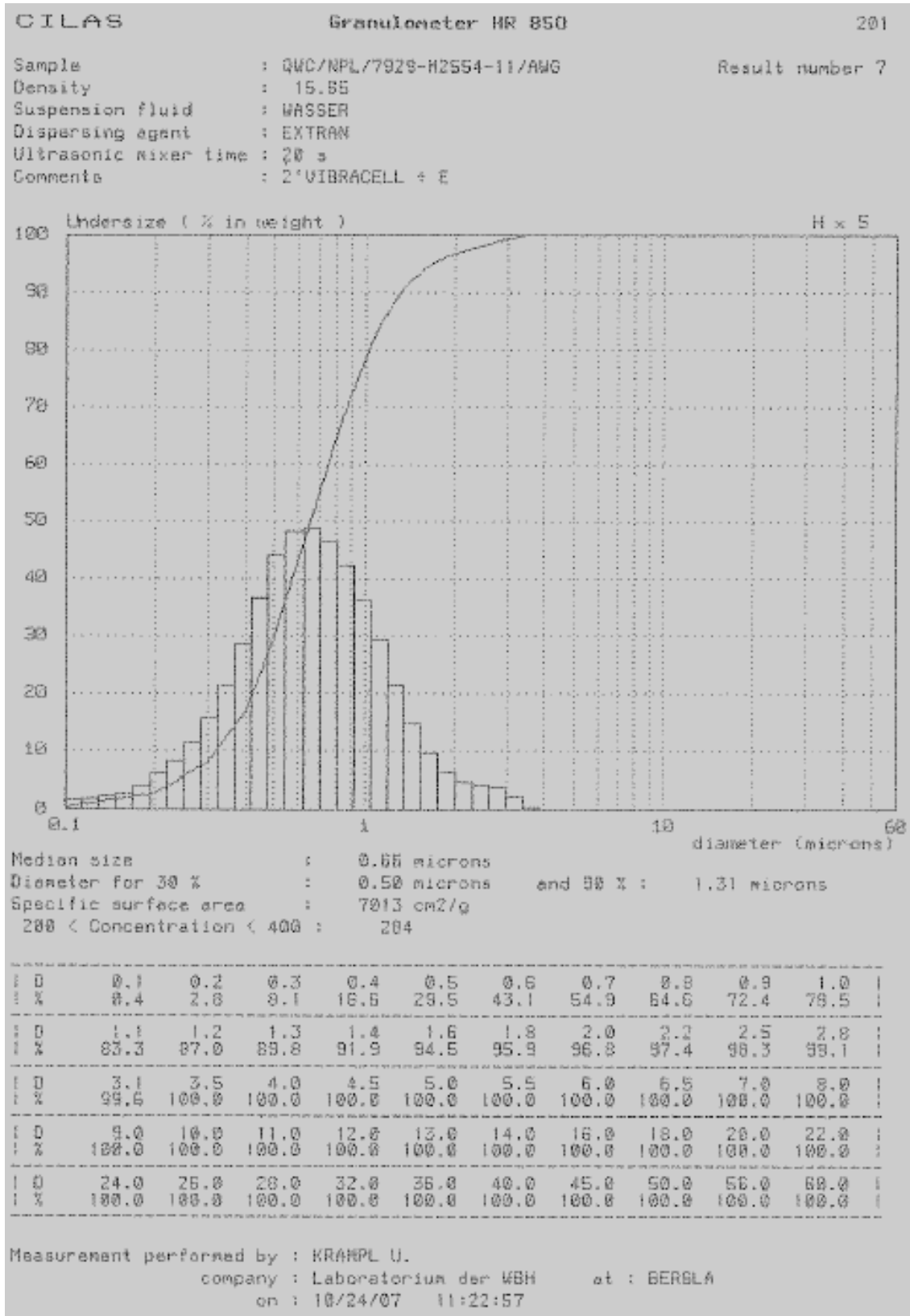


Fig 34 CILAS laser diffusion data for powder SHM 004.

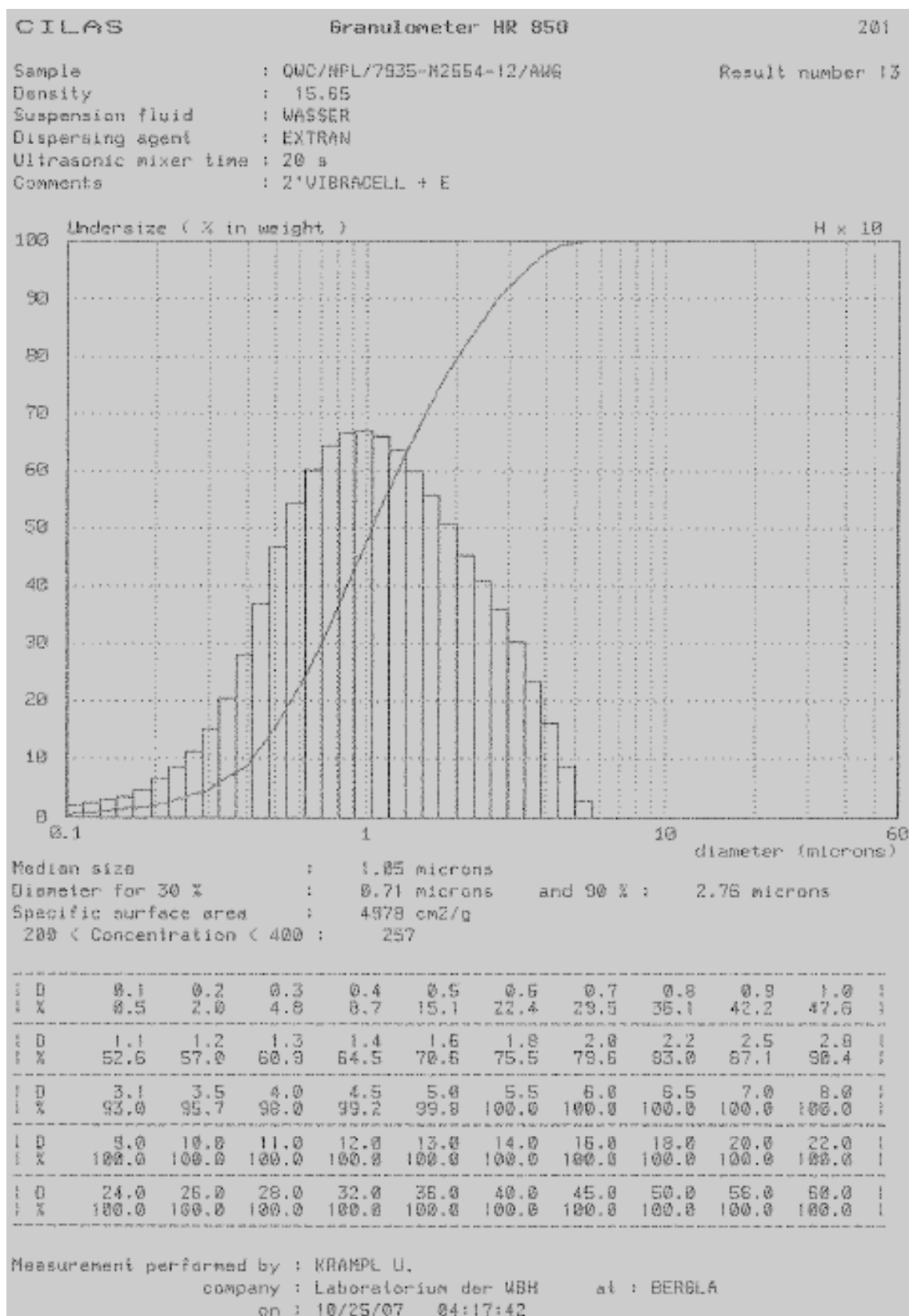


Fig 35 CILAS laser diffraction data for powder SHM 008.

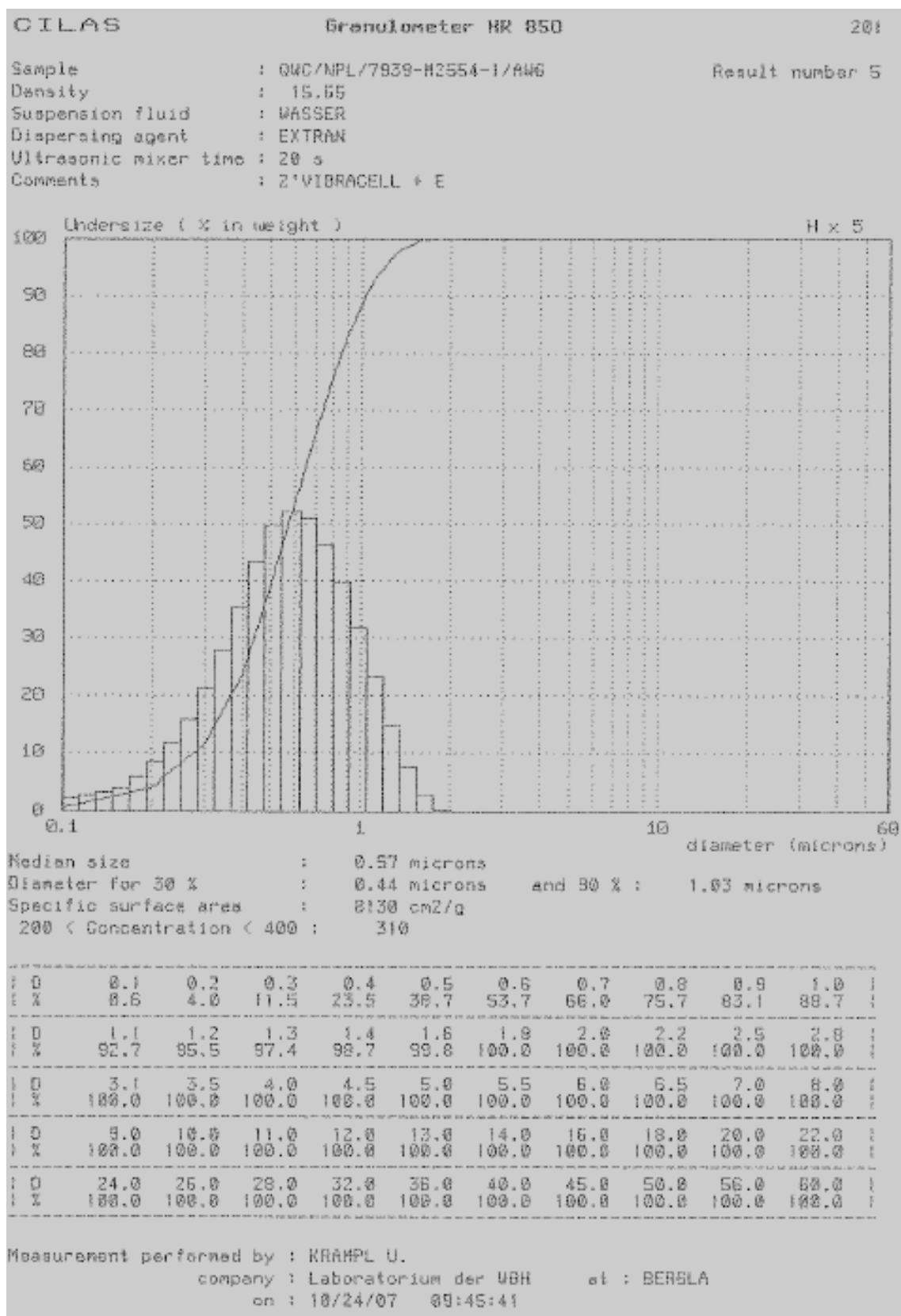


Fig 36 CILAS laser diffusion data for powder GTP BC04.

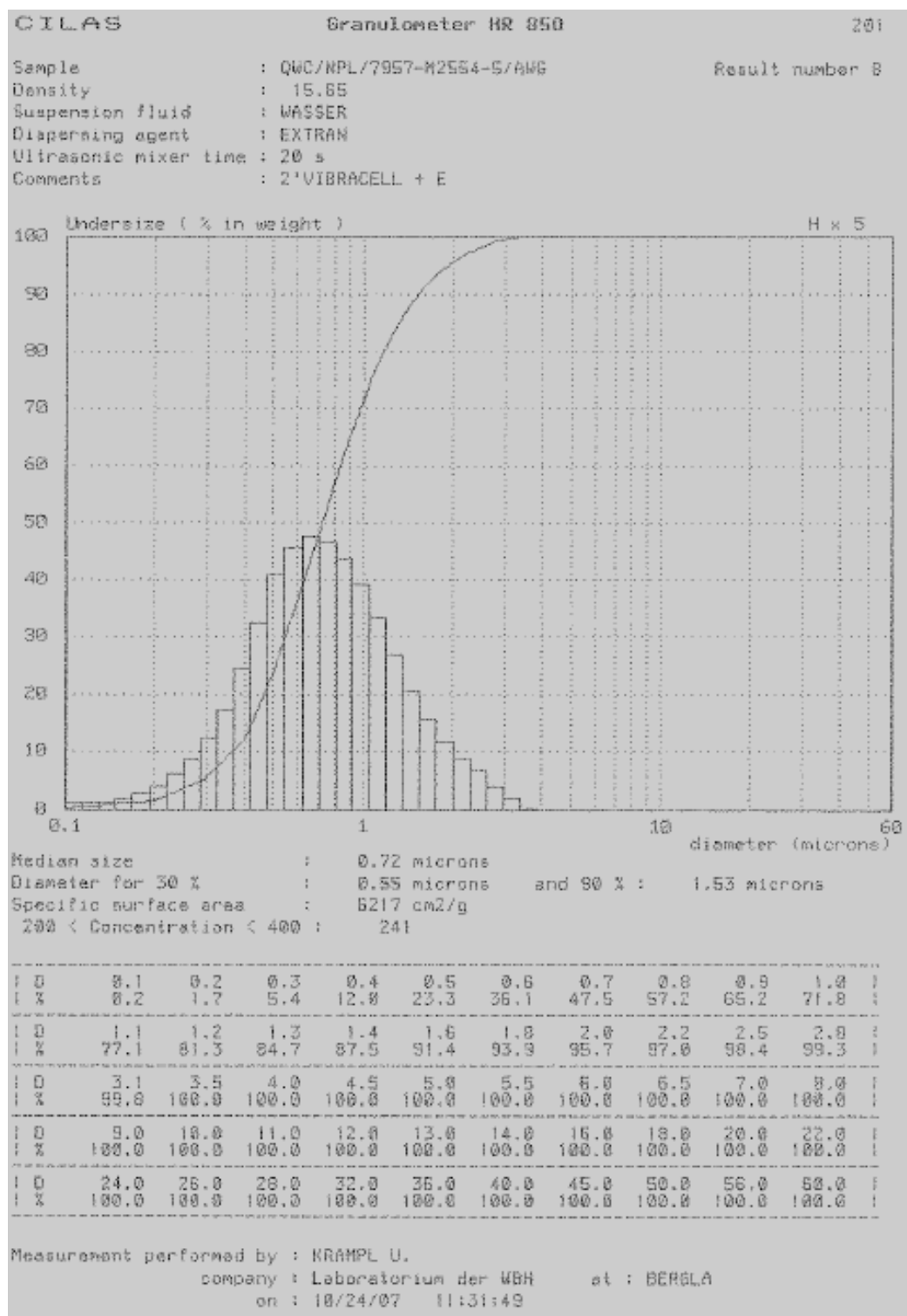


Fig 37 CILAS laser diffusion data for powder GTP S04C.

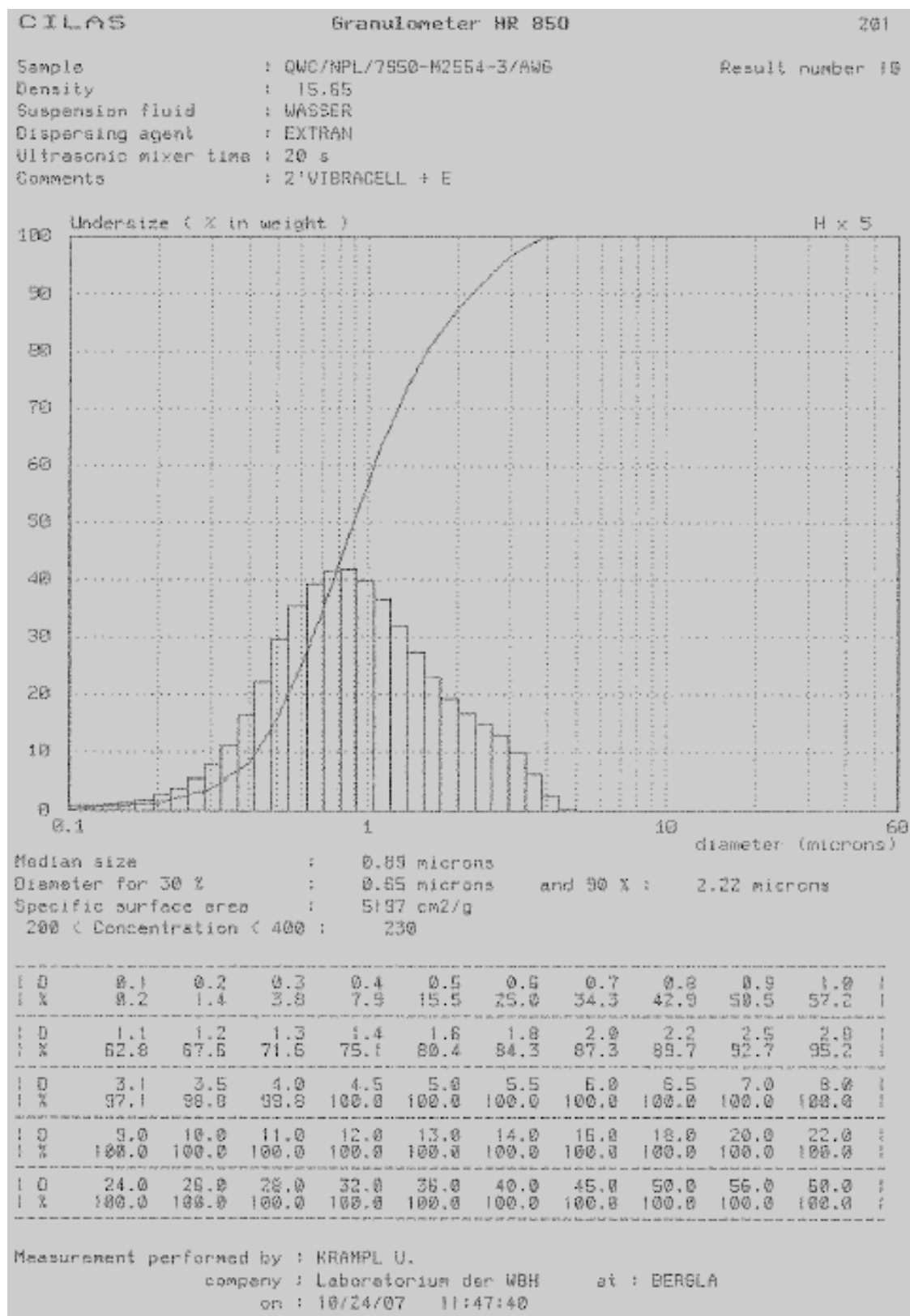


Fig 38 CILAS laser diffusion data for powder GTP S07V.

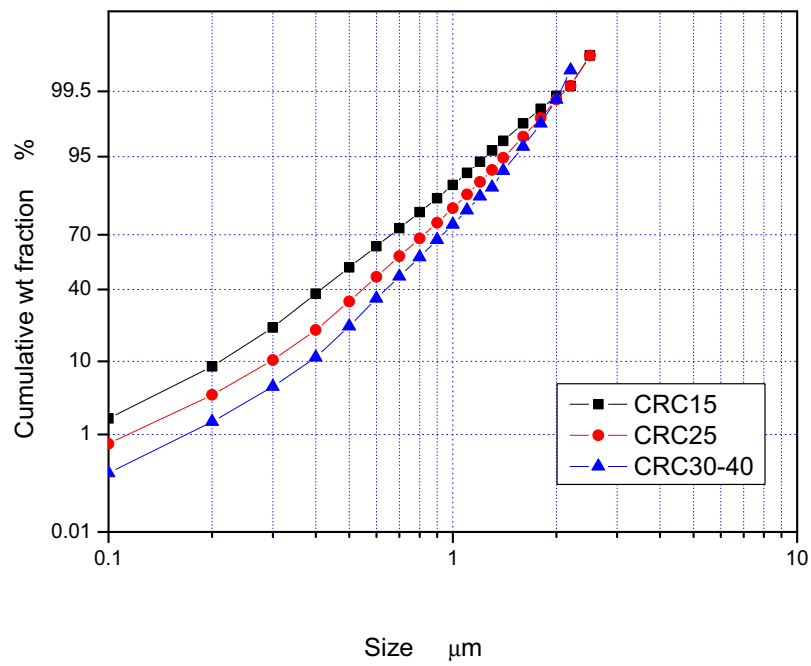


Fig 39 Comparative CILAS data for WBH powders (cumulative).

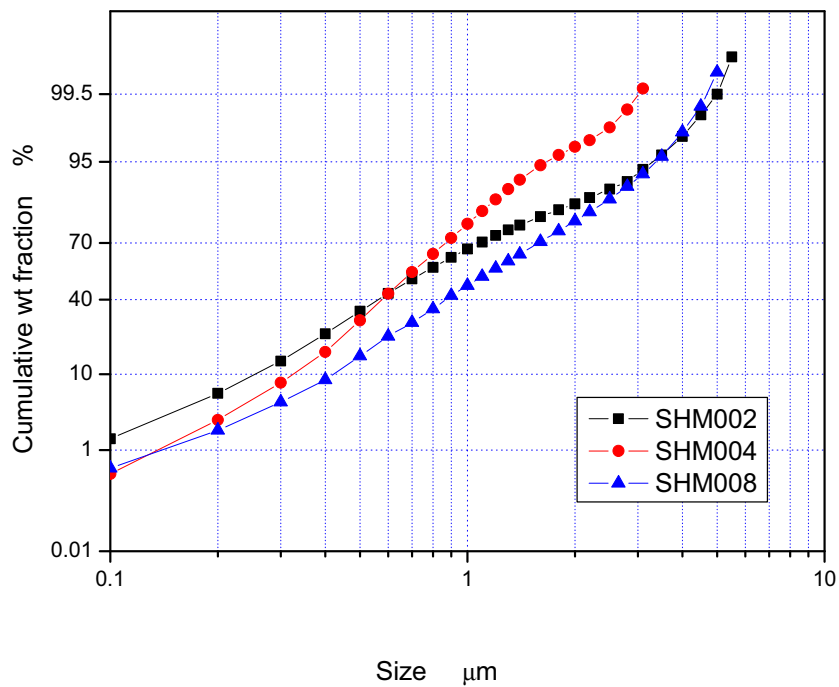


Fig 40 Comparative CILAS data for SHM powders (cumulative).

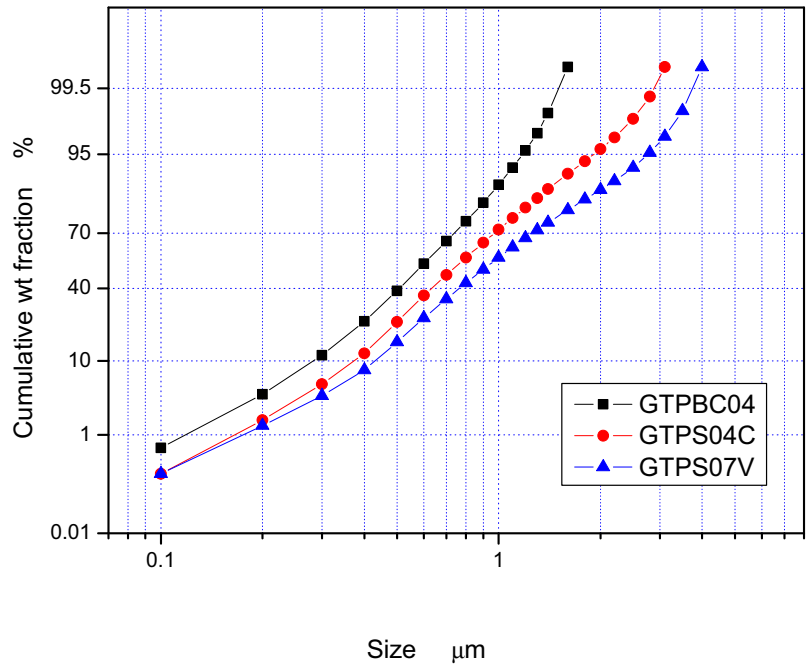


Fig 41 Comparative CILAS data for GTP powders (cumulative).

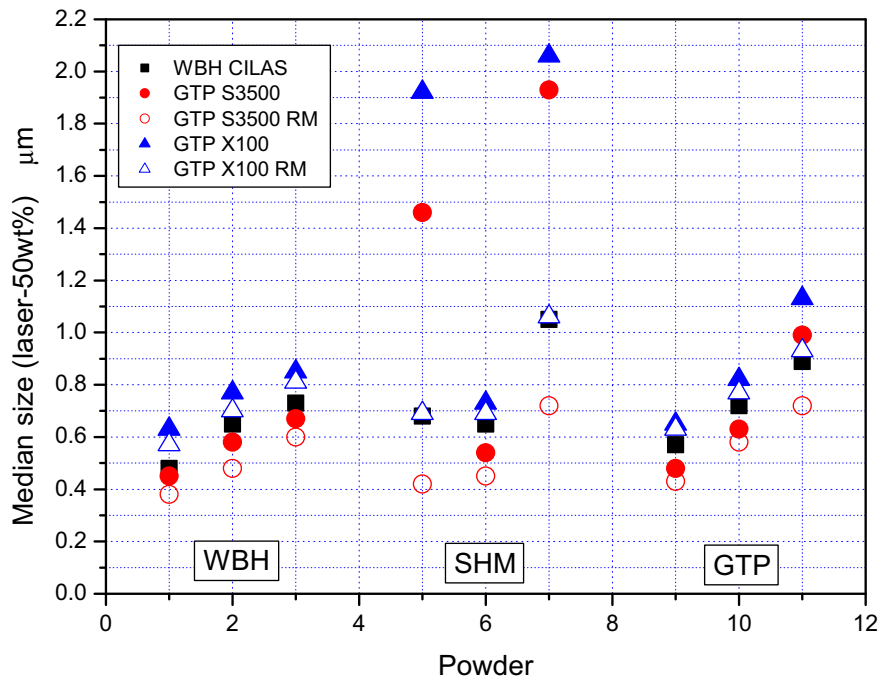


Fig 42 Comparison of median size (50 wt%) values for all laser method measurements.

Note: Following the interlaboratory measurements it was brought to the participants attention that the FSSS measurement systems were no longer to be supplied or maintained by the traditional source. However, a new FSSS equipment supplier, HEL Ltd, (<http://www.helgroup.com/home/about.html>), established contact with the interlab participants and offered to measure the nine powders with their new instrument. The results of this set of measurements are given in Appendix E. The results compared well with the interlab data but there was clearly an issue associated with compaction pressure. Ensuring that this parameter is consistent with established practice will be essential in obtaining reproducible results from this new instrument.

5 INTERLABORATORY DATA – SUMMARY

In the introduction a graph (Fig 1) was presented of the differences in particle size obtained in measurements by Osram Sylvania (GTP) using different techniques. The equivalent graph for the current wider set of measurements examining interlab variations (reproducibility) is shown in Fig 43. The approximate standard deviations associated with lab to lab measurements for the different techniques were not plotted in Fig 43 to keep the figure simple to study. They were:

BET	$\pm 0.02 \mu\text{m}$
FSSS	$\pm 0.05 \mu\text{m}$
Laser	$\pm 0.10 \mu\text{m}$

and are in line with the conclusions of the Osram Sylvania internal study (1).

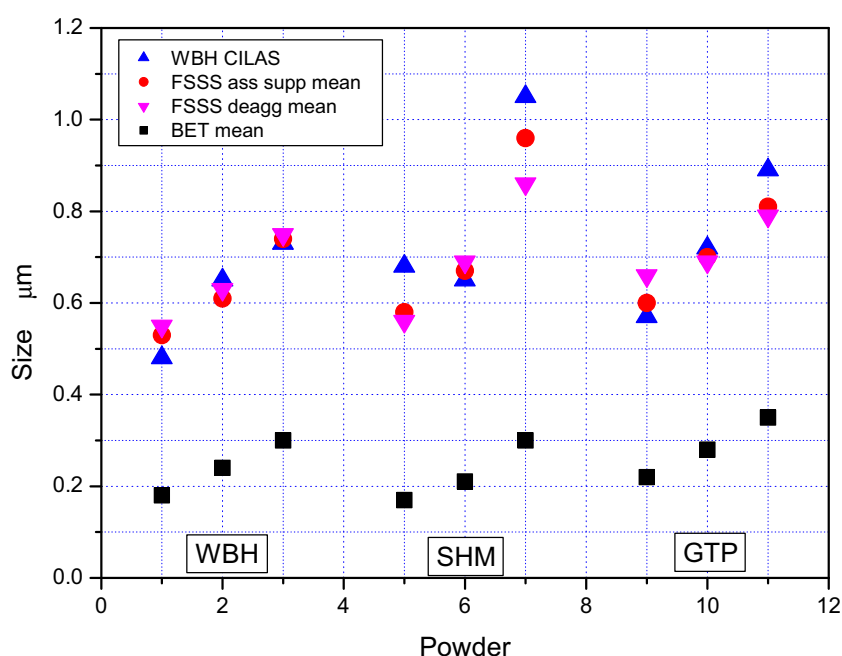


Fig 43 Comparison of particle mean sizes from BET, Laser and FSSS techniques.

6 SUMMARY

An interlaboratory exercise was conducted with participation by three global WC powder suppliers; Sandvik Hard Materials, UK; Wolfram Bergbau -und Hütten GmbH, Austria; and Osram Sylvania, USA (now Global Tungsten Powders) in order to compare powder characterisation techniques based on the sieving, laser and BET methods. The measurements were complemented by oxidation kinetic experiments and high resolution SEM images of the powders (including the use of Orientation Imaging methods) at NPL. There was good reproducibility for the measurements by the three interlab participants, but the three different methods gave significantly different results. Only the BET method gave mean values that corresponded with direct observation by SEM, and this technique is limited by its lack of information on size distribution. Thus, data from the use of these techniques should be treated with caution when using powder sizing data to predict the final sintered structures of WC based hardmetals. There is clearly scope for further research to identify new instrumental approaches to the measurement of WC powder size and distribution in the sub 0.5 micrometre range.

7 ACKNOWLEDGEMENTS

Andreas Bock (WBH), Mike Carpenter (SHM), Thomas Jewett and Kris Schwenke (GTP) are thanked for their organisations' contributions to this exercise. Dipak Gohil at NPL is thanked for conducting the isochronal oxidation measurements on the WC powders. Support for NPL coordination was provided by the NPL "Materials Metrology" programme funded by the UK Department of Innovation, Universities and Skills (DIUS), now The Department of Business, Innovation and Skills (BIS).

8 REFERENCES

1. G.K. Schwenke and F.J. Venskytis. Particle Size Analysis of Sub-Micrometre WC Powders. MPIF Int. Conf. on PM Materials, Cemented Carbide Symposium, June 2004, Chicago, USA.
2. B. Roebuck, E.G. Bennett, E.A. Almond and M.G. Gee. Dependence of Oxidation Rate of WC Powder on Particle Size, *J. Mater. Sci.* 1986, **21**, 2033-2042.
3. B. Roebuck, E.A. Almond and J.L.F. Kellie. Characterisation of the Particle Size of Cubic Transition Metal Carbides by Measurement of the Rate of Oxidation, PM 86, EPMF, Dusseldorf, West Germany, July 1986, Horizons of Powder Metallurgy, eds. W.A. Kaysser and W.J. Huppman. Pt I, 123-126.
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5. K.P. Mingard, B. Roebuck, E.G. Bennett, M.G. Gee, H. Nordenstrom, G. Sweetman and P. Chan. Comparison of EBSD and Conventional Methods of Grain Size Measurement of Hardmetals, 9th Int. Conf. Science of Hard Materials, Jamaica, March 2008, and *Int. J. Refractory Metals and Hard Materials*, 2009, **27**(2), 213-223.
6. K. Mannesson, M. Elfving, A. Kussofsky, S. Norgren and J. Agren, "Analysis of WC grain growth during sintering using EBSD and image analysis", *Int. J. Refractory Metals and Hard Materials*, 2008, **26**, 449-455.

APPENDIX A
POWDER SOURCE DATA SUPPLIED BY
MANUFACTURERS

Wolfram Bergbau- und Hütten GmbH (WBH)
Sandvik Hard Materials (SHM)
Global Tungsten Powders – GTP (Osram Sylvania)

wolfram**Bergbau- und Hütten-GmbH Nfg. KG**

A-8542 St. Peter i. S./Austria, Tel: +43 (0)3465/7077-0, Fax: -10

INSPECTION CERTIFICATE

FO-101/Rev.2 acc. to EN10204 3.1 (DVR 0086991)

Dr. Bryan Roebuck, FIMM CEng
 Materials Centre
 Physical Laboratory

Teddington
 TW11 0LW MIDDLESEX

Contract No.: -
 Delivery Note: 407703 11.10.2006
 Quantity: 6 kg Date: 11.10.2006



Dipl.Ing.(FH) A.Zierler
 Authorized Representative

Information: 6 x 1 kg

Material: CRC015U

Version: 1

Lot No.: 376M06WC

PHYSICAL ANALYSIS

	BET-Surface:	2.31	m ² /g
Grain size (FSSS) as supplied:		0.52	µm
Grain size (FSSS) lab milled:		0.57	µm
Scott-density:		26	g/inch ³

CHEMICAL ANALYSIS

Ct:	6.09	%
Cf:	0.05	%
Al:	2	ppm
Ca:	5	ppm
Co:	<5	ppm
Fe:	40	ppm
Mo:	<10	ppm
Na:	19	ppm
Ni:	<5	ppm
O:	1420	ppm
S:	<5	ppm
Si:	<10	ppm

PARTICLE SIZE DISTRIBUTION

CILAS Laserdiffraction: Curve enclosed

Attention further page >>>

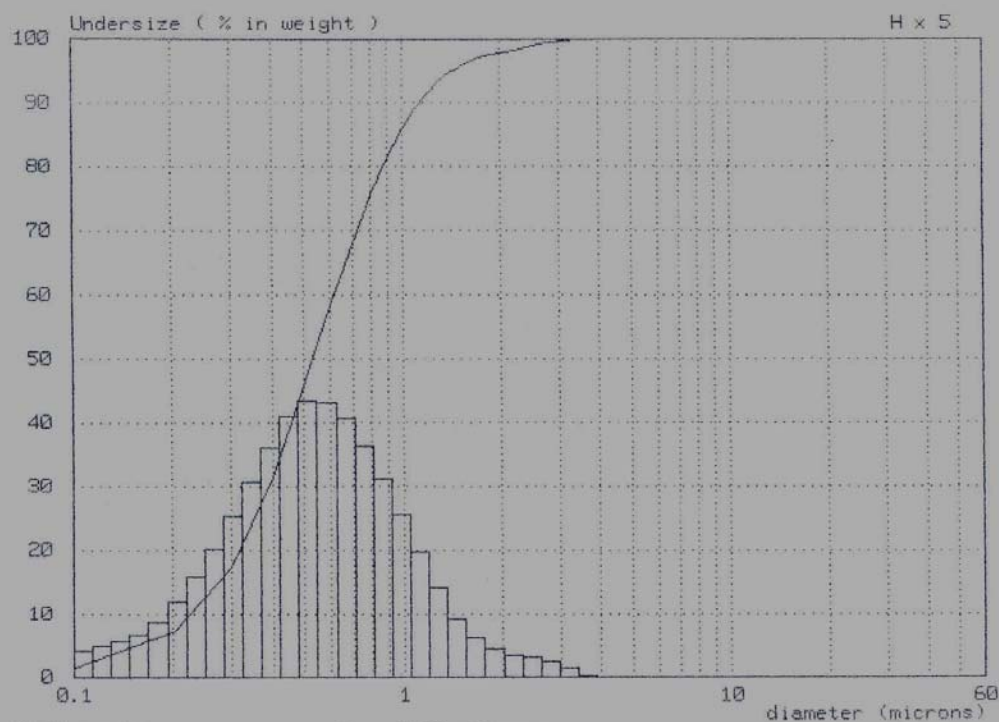
CILAS

Granulometer HR 850

201

Sample : 376/M/06/WCV
 Density : 15.65
 Suspension fluid : WASSER
 Dispersing agent : EXTRAN
 Ultrasonic mixer time : 20 s
 Comments : 2 MIN VC+E

Result number 1



Median size : 0.53 microns
 Diameter for 30 % : 0.39 microns and 90 % : 1.13 microns
 Specific surface area : 9184 cm²/g
 200 < Concentration < 400 : 313

D	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
%	1.4	7.0	17.4	31.5	45.8	58.3	68.1	75.7	81.5	85.8
D	1.1	1.2	1.3	1.4	1.6	1.8	2.0	2.2	2.5	2.8
%	89.1	91.6	93.4	94.7	96.4	97.3	97.9	98.4	99.0	99.5
D	3.1	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	8.0
%	99.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	9.0	10.0	11.0	12.0	13.0	14.0	16.0	18.0	20.0	22.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	24.0	26.0	28.0	32.0	36.0	40.0	45.0	50.0	56.0	60.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Measurement performed by : SAILER
 company : Laboratorium der WBH at : BERGLA
 on : 09/15/06 09:35:20

wolfram

Bergbau- und Hütten-GmbH Nfg. KG
 A-8543 St. Martin i.S./Austria, Tel: +43 (0)3465/7077-0, Fax: -10

INSPECTION CERTIFICATE

FO-101/Rev.2 acc. to EN10204 3.1 (DVR 0086991)

Dr. Bryan Roebuck, FIMM CEng
 Materials Centre
 Physical Laboratory

Teddington
 TW11 1OLW MIDDLESEX

Contract No.: -
 Delivery Note: 407703 11.10.2006
 Quantity: 6 kg Date: 04.12.2007


 Dipl.-Ing. (FH) A. Zierler
 Authorized Representative

Information: 6 x 1 kg

Material: CRC025U

Version: 1

Lot No.: 336M05WC

PHYSICAL ANALYSIS

BET-Surface:	1.69	m ² /g
Grain size (FSSS) as supplied:	0.66	µm
Grain size (FSSS) lab milled:	0.70	µm
Scott-Density:	28	g/inch ³

CHEMICAL ANALYSIS

Cf:	6.12	%
Cf:	0.04	%
Al:	1	ppm
Ca:	<2	ppm
Co:	<5	ppm
Fe:	29	ppm
Mo:	<10	ppm
Na:	14	ppm
Ni:	<5	ppm
O:	1150	ppm
S:	<5	ppm
Si:	<10	ppm

PARTICLE SIZE DISTRIBUTION

CILAS Laserdiffracton: Curve enclosed

Attention further page >>>

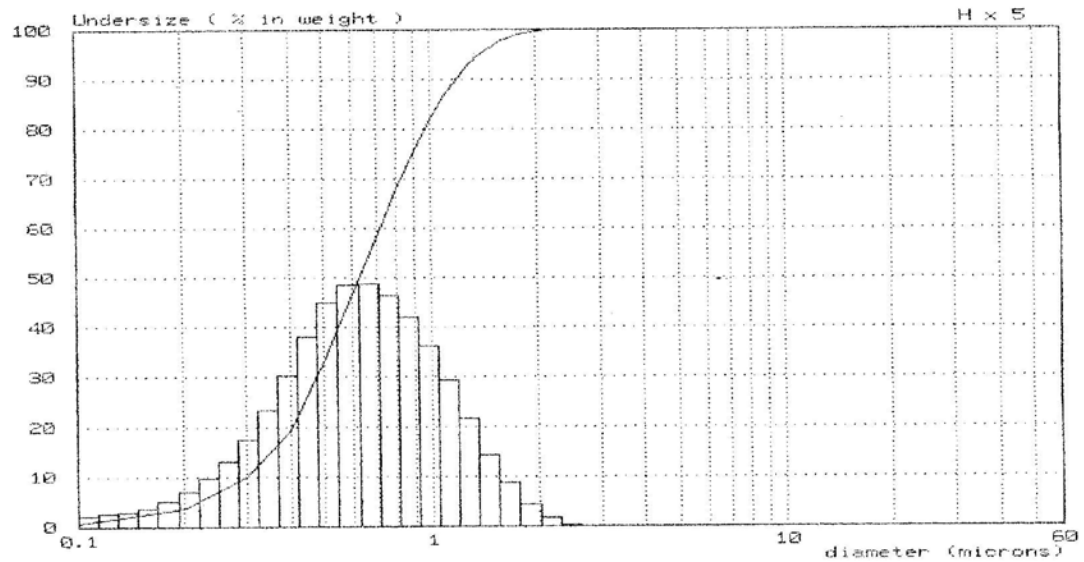
CILAS

Granulometer HR 850

201

Sample : 338/M/05/WCV/CRC025U
 Density : 15.65
 Suspension fluid : WASSER
 Dispersing agent : EXTRAN
 Ultrasonic mixer time : 20 s
 Comments : 2 MIN VC + E

Result number 2



Median size : 0.63 microns
 Diameter for 30 % : 0.48 microns and 90 % : 1.19 microns
 Specific surface area : 7537 cm²/g
 200 < Concentration < 400 : 335

D	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
%	0.6	3.6	9.9	19.6	32.8	46.5	58.3	67.9	75.7	81.9
D	1.1	1.2	1.3	1.4	1.6	1.8	2.0	2.2	2.5	2.8
%	86.7	90.4	93.2	95.3	97.8	99.1	99.7	99.9	100.0	100.0
D	3.1	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	8.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	9.0	10.0	11.0	12.0	13.0	14.0	16.0	18.0	20.0	22.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	24.0	26.0	28.0	32.0	36.0	40.0	45.0	50.0	55.0	60.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Measurement performed by : KRAUS
 company : Laboratorium der WBH at : BERGLA
 on : 09/12/05 08:15:50

wolfram**Bergbau- und Hütten-GmbH Nfg. KG**

A-8542 St. Peter i. S./Austria, Tel: +43 (0)3465/7077-0, Fax: -10

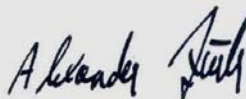
INSPECTION CERTIFICATE

FO-101/Rev.2 acc. to EN10204 3.1 (DVR 0086991)

Dr. Bryan Roebuck, FIMM CEng
Materials Centre
Physical Laboratory

Teddington
TW11 OLW MIDDLESEX

Contract No.: -
Delivery Note: 407703 11.10.2006
Quantity: 6 kg Date: 11.10.2006


Dipl.Ing.(FH) A.Zierler
Authorized Representative

Information: 6 x 1 kg

Material: CRC030-40

Version: 1

Lot No.: 75405WC

PHYSICAL ANALYSIS

Grain size (FSSS) as supplied:	0.80	µm
Grain size (FSSS) lab milled:	0.84	µm
Scott-Density:	30	g/inch ³

CHEMICAL ANALYSIS

Ct:	6.13	%
Cf:	0.03	%
Al:	1	ppm
Ca:	2	ppm
Co:	<5	ppm
Fe:	43	ppm
Mo:	10	ppm
Na:	18	ppm
Ni:	6	ppm
O:	880	ppm
S:	<5	ppm
Si:	<10	ppm
doped with Cr ₃ C ₂ :	#	%
doped with VC:	#	%
doped with TaC:	#	%

PARTICLE SIZE DISTRIBUTION

CILAS Laserdiffraction: Curve enclosed

Attention further page >>>

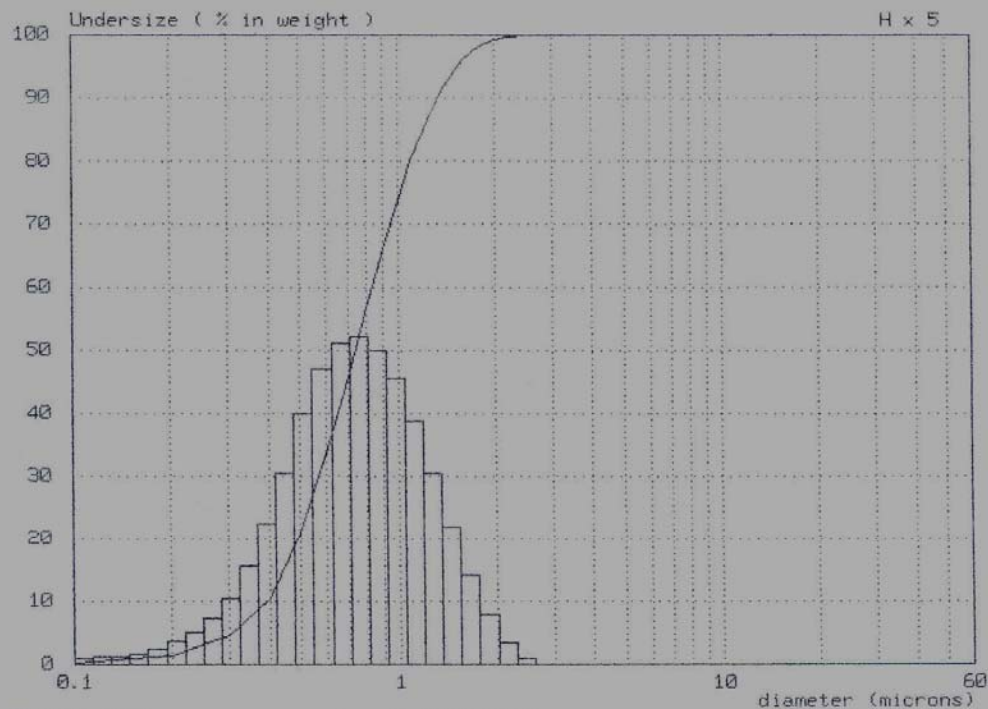
CILAS

Granulometer HR 850

201

Sample : 754/05/WCV
 Density : 15.65
 Suspension fluid : WASSER
 Dispersing agent : EXTRAN
 Ultrasonic mixer time : 20 s
 Comments : 2MIN VC+E

Result number 5



Median size : 0.74 microns
 Diameter for 30 % : 0.57 microns and 90 % : 1.33 microns
 Specific surface area : 6122 cm²/g
 200 < Concentration < 400 : 330

D	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
%	0.2	1.5	4.7	10.3	20.8	33.7	45.9	56.8	65.0	73.7
D	1.1	1.2	1.3	1.4	1.6	1.8	2.0	2.2	2.5	2.8
%	80.0	85.0	88.9	92.0	96.0	98.2	99.3	99.8	100.0	100.0
D	3.1	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	8.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	9.0	10.0	11.0	12.0	13.0	14.0	16.0	18.0	20.0	22.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
D	24.0	25.0	28.0	32.0	35.0	40.0	45.0	50.0	55.0	60.0
%	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0



CERTIFICATE OF QUALITY

No. : 249-2007

Tungsten Carbide Powder

Customer: **Dr Bryan Roebuck**
 FIMMM CEng Materials Centre
 Division of Engineering and Process Control (DEPC)
 National Physical Laboratory, Teddington,
 Middlesex
 TW11 0LW

Anal. No.	:	3124/07
Batch No.	:	BC04U7012
Mass (kg)	:	10

Parameter	Unit	Results of inspection
Fisher grain size as delivered	(μm)	0,63
Fisher porosity as delivered		0,646
Fisher grain size lab milled	(μm)	0,63
Fisher porosity lab milled		0,621
Scott density	(g.cm-3)	2,40
Tap density	(g.cm-3)	3,73
O2	(%)	0,188
C total	(%)	6,11
C free	(%)	0,01
Al	(ppm)	10
Ca	(ppm)	2
Co	(ppm)	6
Cr	(ppm)	24
Cu	(ppm)	2
Fe	(ppm)	135
K	(ppm)	2
Mg	(ppm)	<1
Mn	(ppm)	4
Mo	(ppm)	9
Na	(ppm)	3
Ni	(ppm)	11
Pb	(ppm)	<5
S	(ppm)	2

Date of issue : 13.3.2007

OSRAM Bruntál spol. s r.o.
 Zahradní 46 • CZ - 79201 Bruntál

OSRAM  13
 Ing. Jiří Šulc
 Laboratory Manager

Specified results of analyses are related to mentioned batch only.
 The certificate of quality cannot be copied without an agreement of
 laboratory unlike whole one.



PARTICLE SIZE DISTRIBUTION

CILAS 1064 Liquid

Range : 0.04 μ m - 500.00 μ m / 100 Classes

Sample Ref : WC 3124/BC04U7012 lm
 Type produit : WC
 Client : OSRAM Bruntál spol. s r. o.
 Comments : US during meas., 50W
 Liquid : water
 Dispersing agent : HMF
 Operator : Kunovska
 Company : OSRAM Bruntál spol. s r. o.
 Location : laboratoř
 Date : 07.03.2007 Time : 15:43:35
 Measurement number 12732

Ultrasounds : 100 s (+during)
 Concentration : 115
 Diameter at 10% : 0.09 μ m
 Diameter at 50% : 0.40 μ m
 Diameter at 90% : 1.04 μ m
 Fraunhofer
 Density/Factor : -----
 Specific surface : -----
 Auto. dilution/top up : Yes / No
 Nb Measur./Rins. : 20 / 4

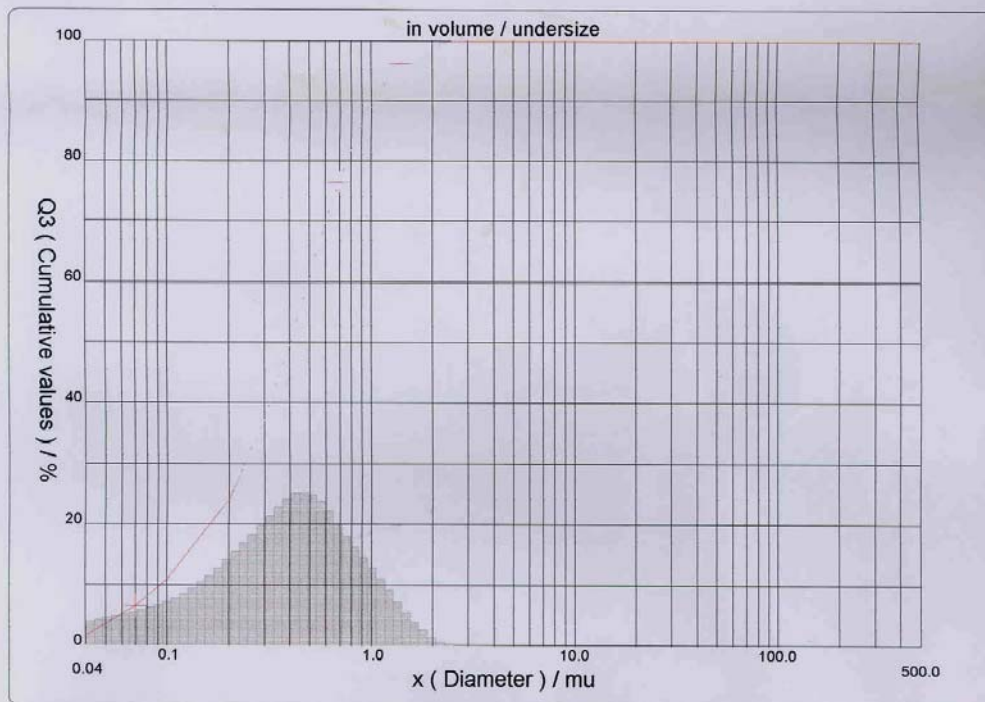
Customer defined classes

in volume / undersize

x	0.10	0.50	0.70	0.90	1.20	1.50	1.80	2.10	2.50	2.90
Q3	10.82	60.53	76.53	85.77	93.44	97.24	98.96	99.67	99.93	100.00

x	3.50	4.00	4.50	5.00	5.50	6.00	7.00	10.00	20.00	100.0
Q3	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.0

x : diameter / μ m Q3 : cumulative value / % q3 : population density / %



Serial nb : 264

Ref : 1.r9.m0.45M1818/3.30/12732/m6.20.5.10.1Fh.20.5.10.1FhVQ-0.0.0.0/300.0.15.g60.15.9.10.1.10.N.1.10.N.0V.1.15

APPENDIX B

FEGSEM IMAGES

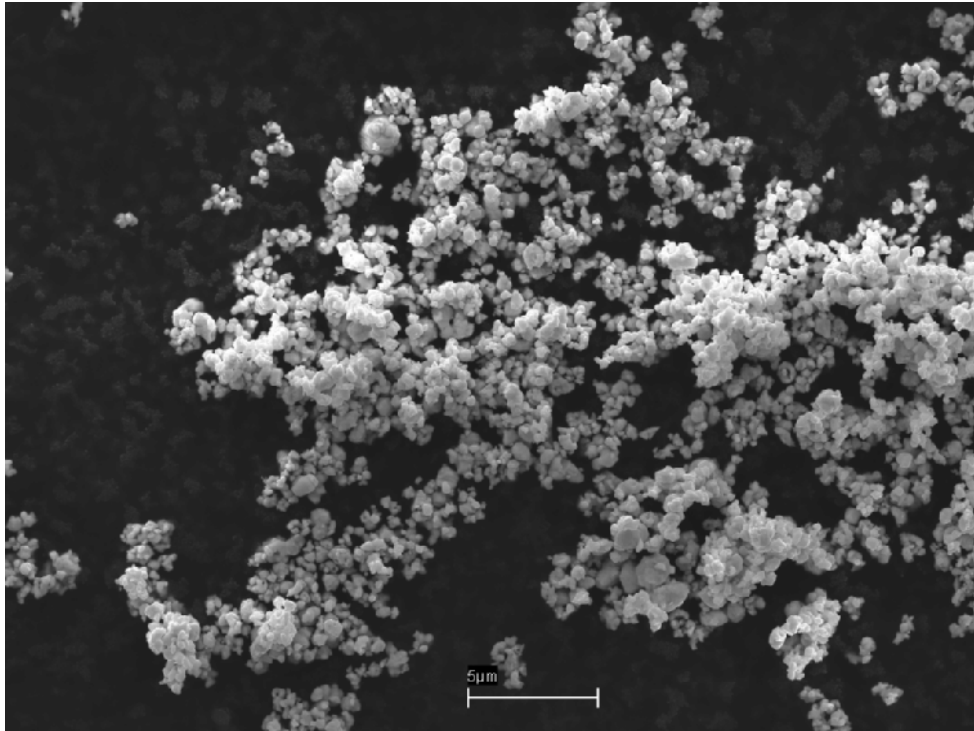


Fig B1 WBH – CRC030: 10k nominal magnification

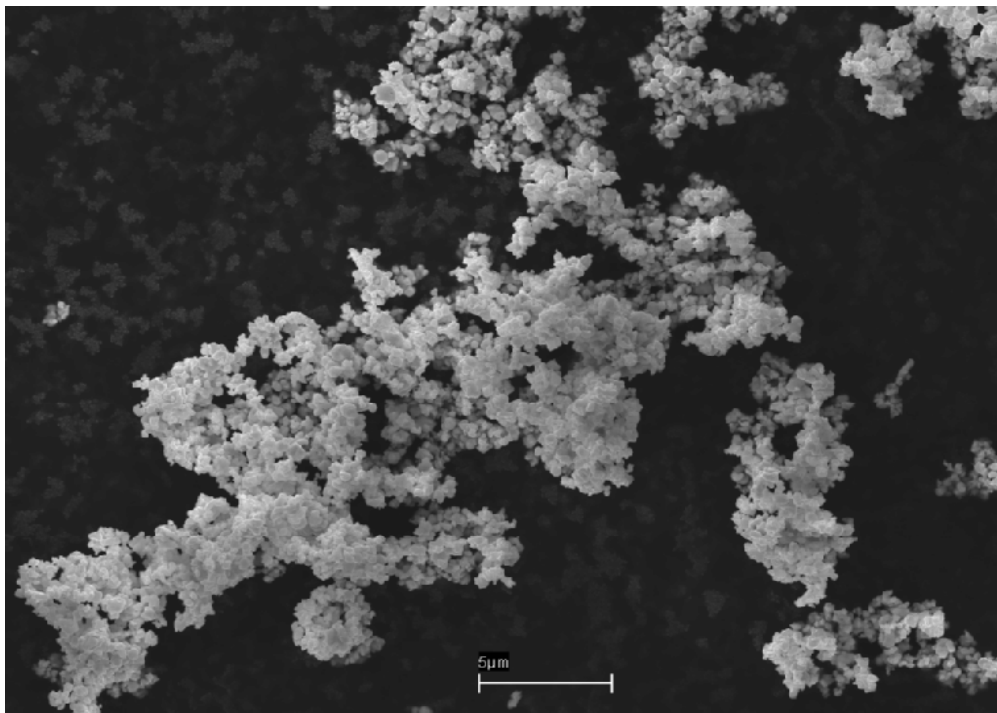


Fig B2 WBH - CRC025; 10k nominal magnification

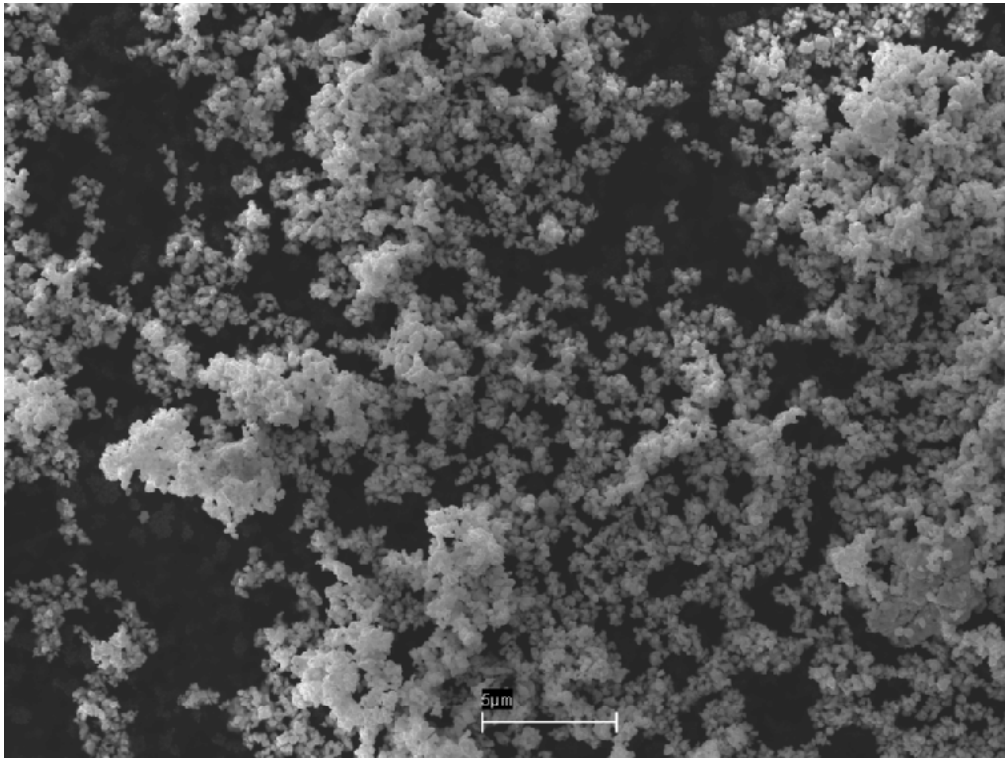


Fig B3 WBH – CRC015: 10k nominal magnification

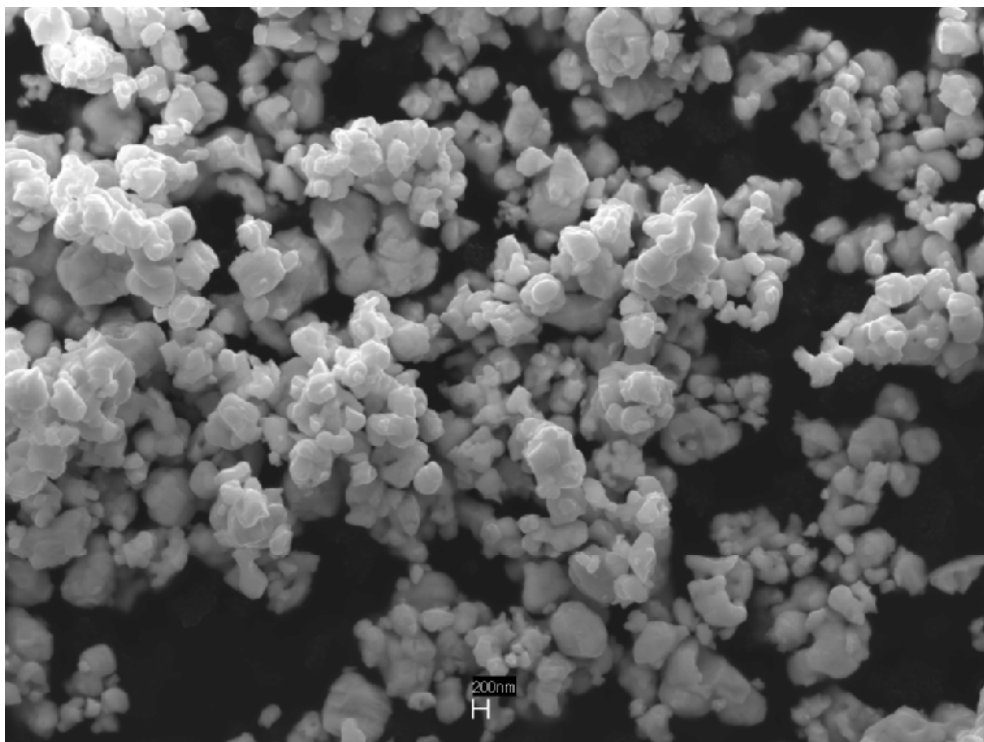


Fig B4 WBH - CRC030; 30k nominal magnification

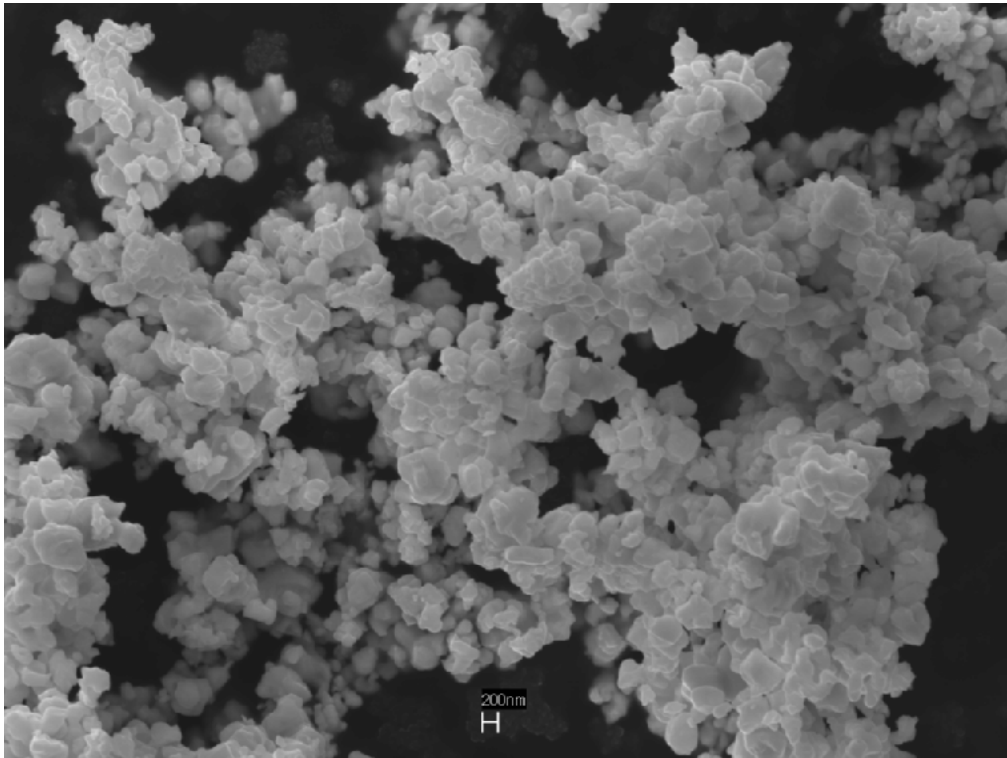


Fig B5 WBH – CRC025: 30k nominal magnification

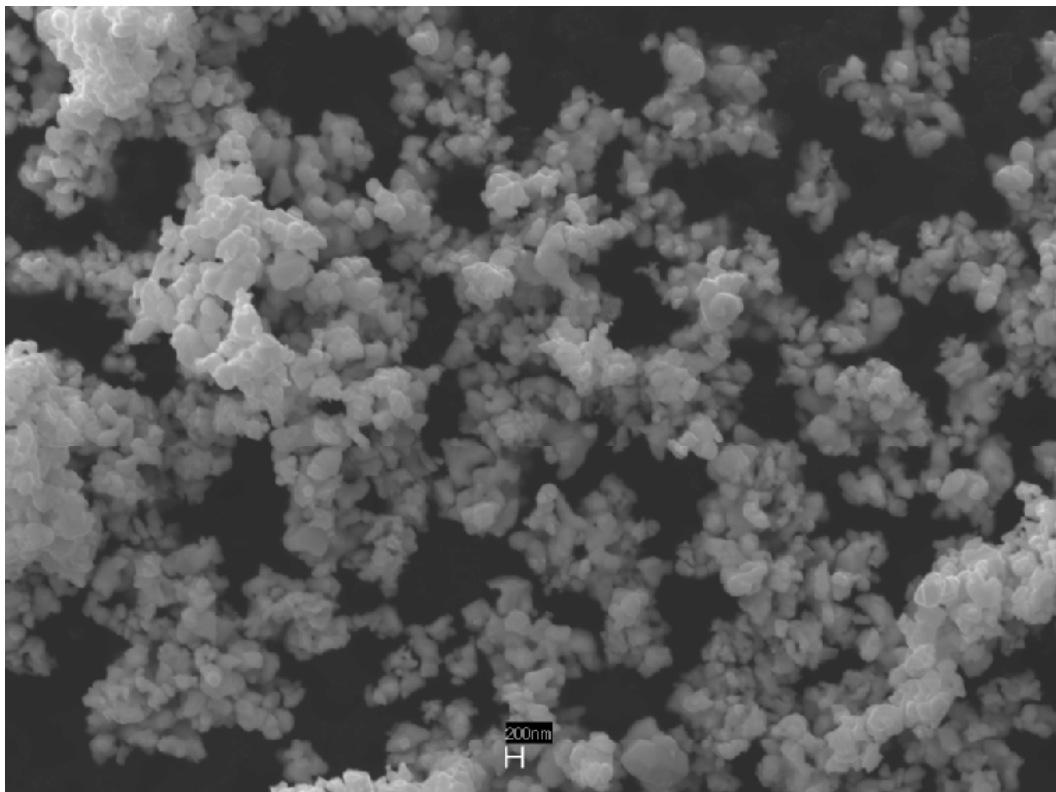


Fig B6 WBH - CRC015; 30k nominal magnification

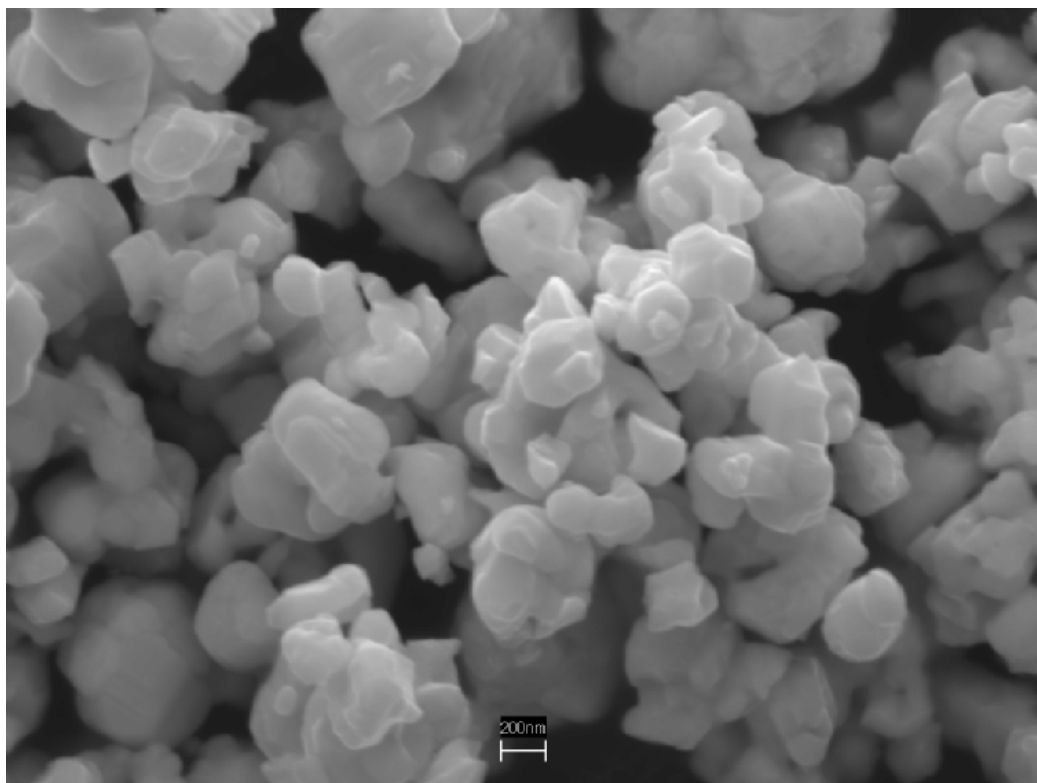


Fig B7 WBH - CRC030; 80k nominal magnification

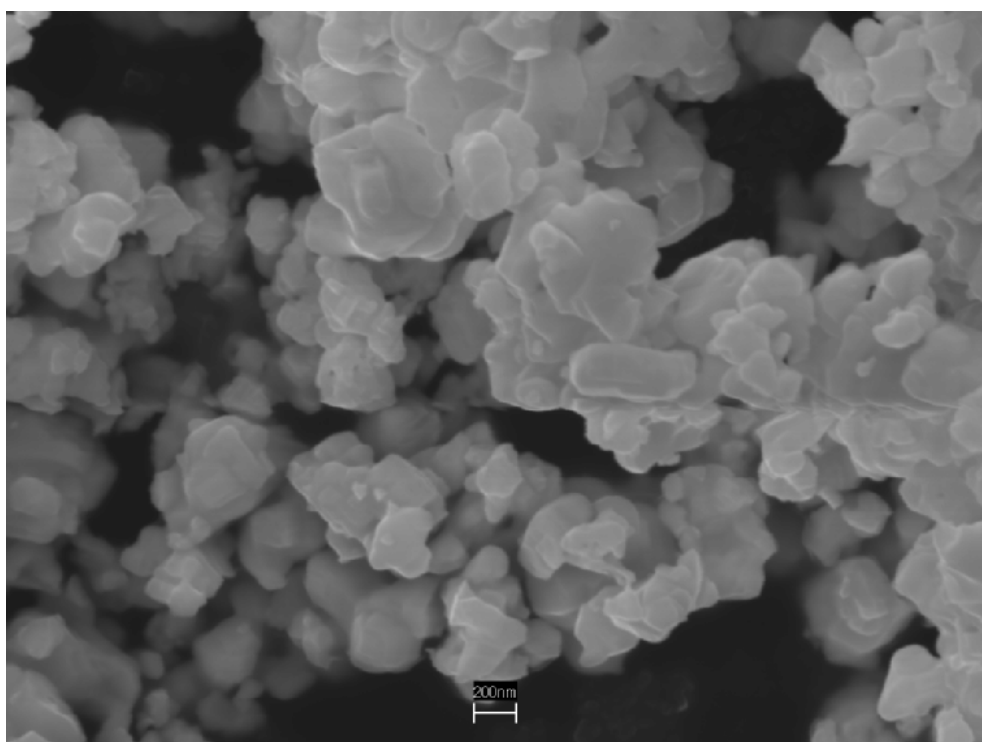


Fig B8 WBH - CRC025; 80k nominal magnification

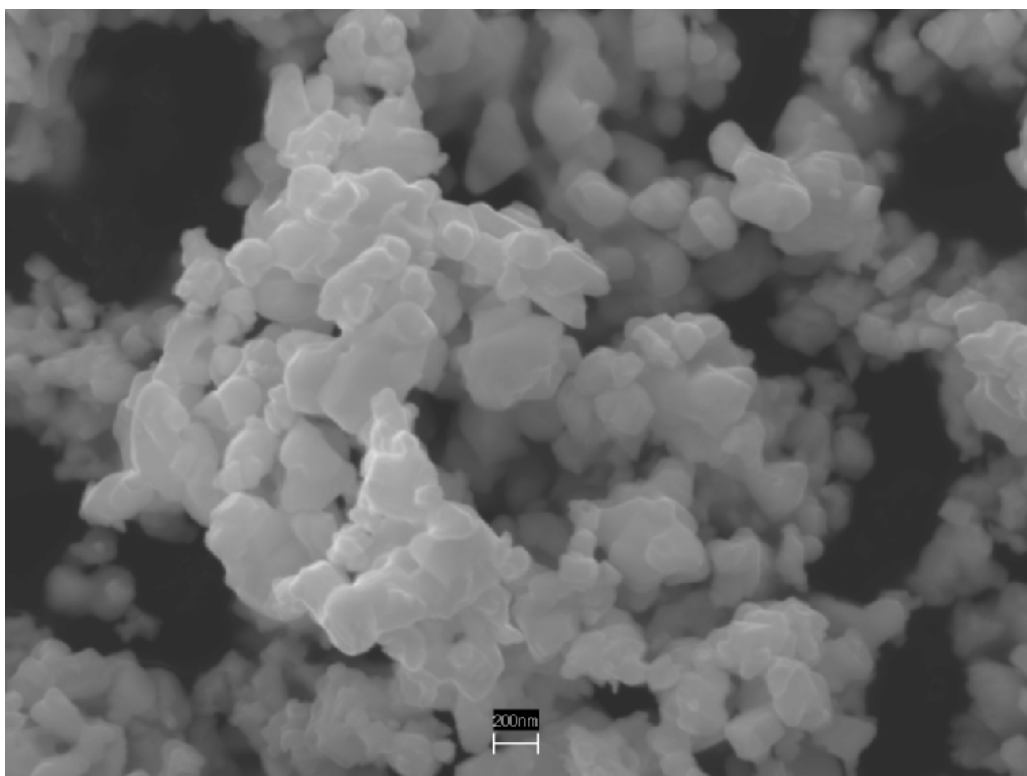


Fig B9 WBH – CRC015: 80k nominal magnification

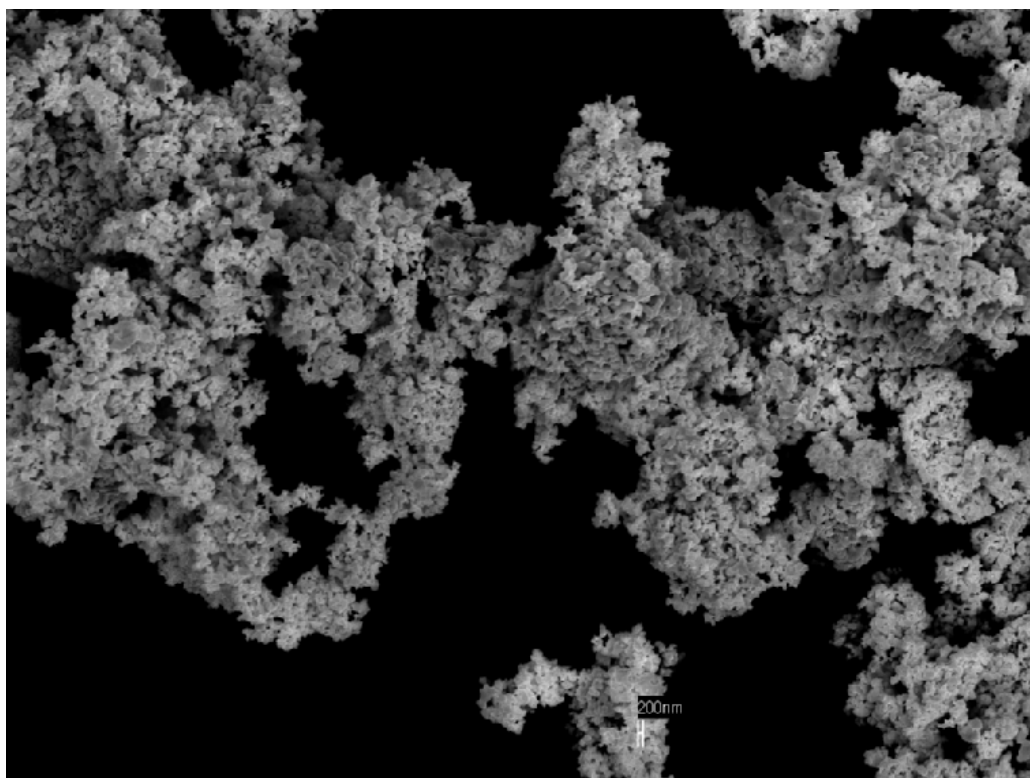


Fig B10 SHM002 - 10k nominal magnification

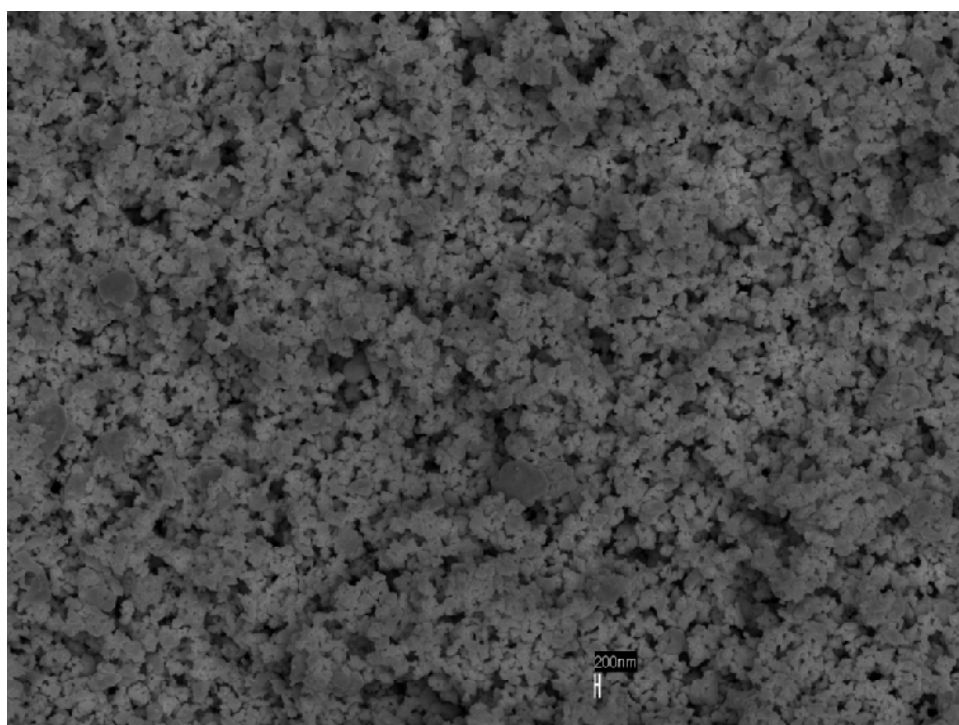


Fig B11 SHM004 - 10k nominal magnification

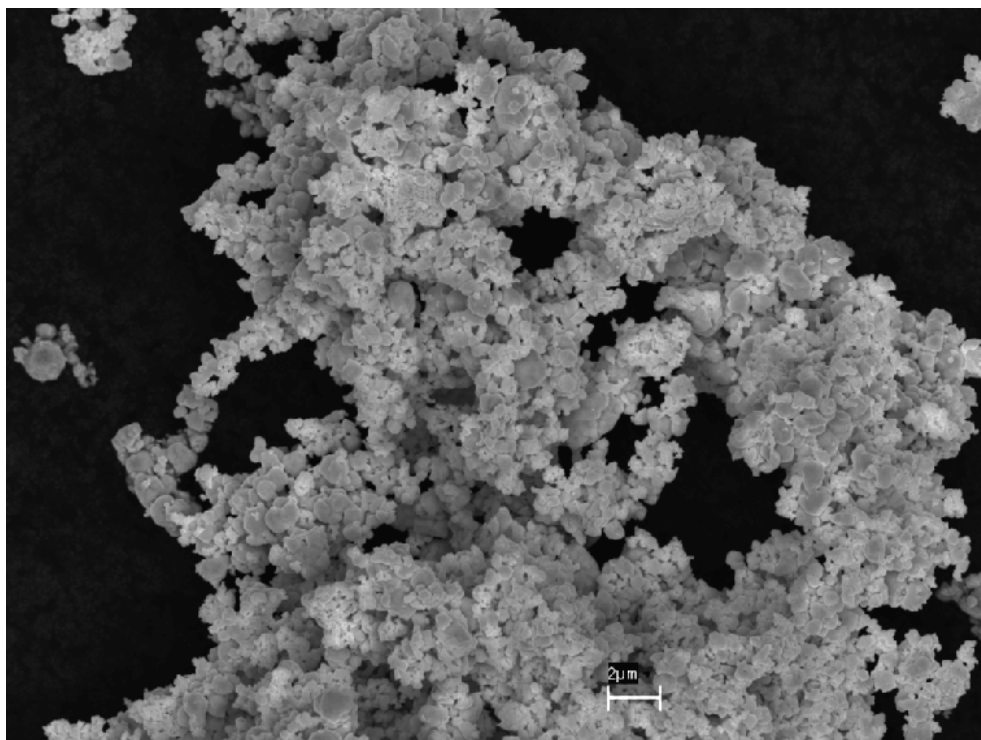


Fig B12 SHM008 - 10k nominal magnification

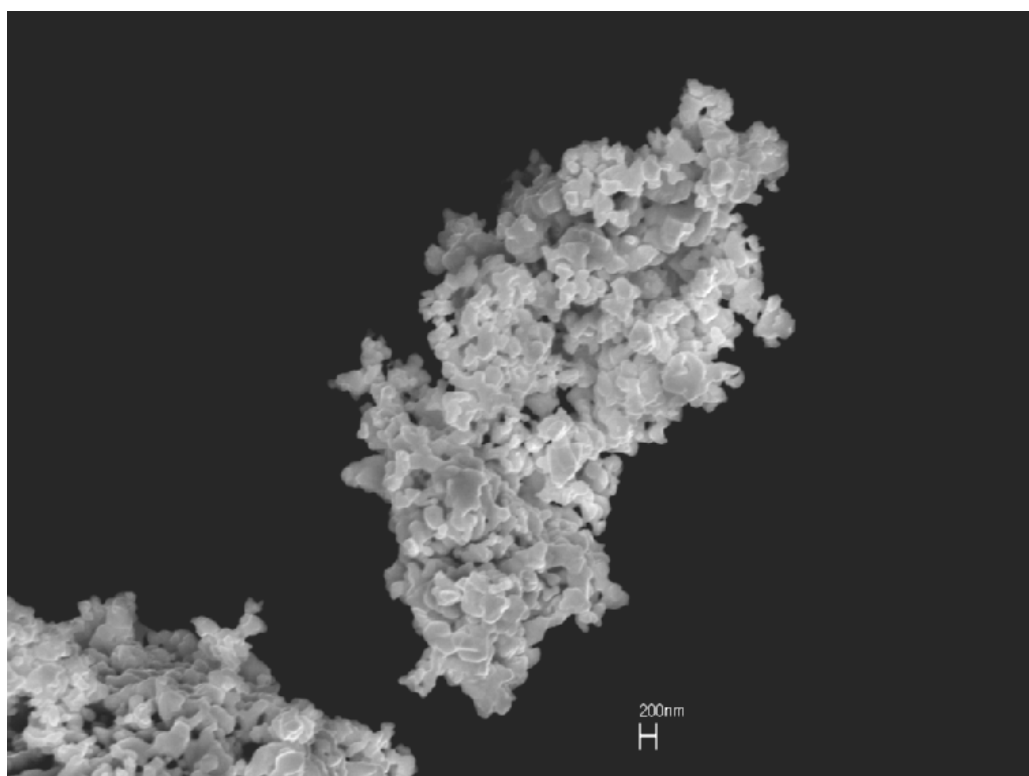


Fig B13 SHM002 - 30k nominal magnification

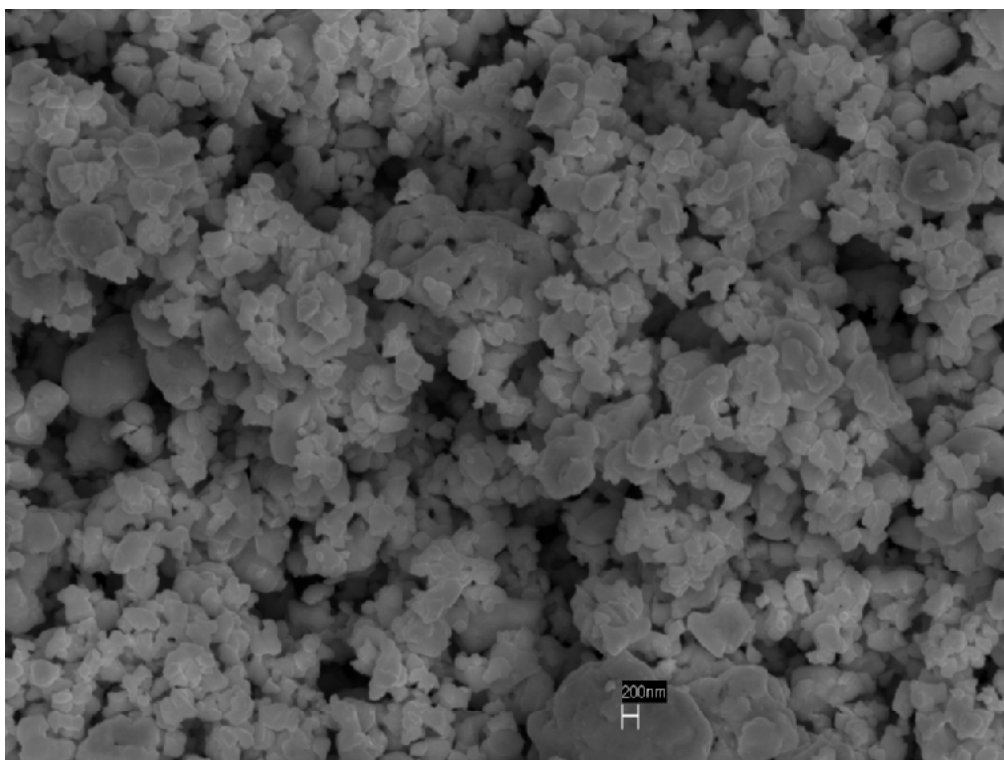


Fig B14 SHM004 - 30k nominal magnification

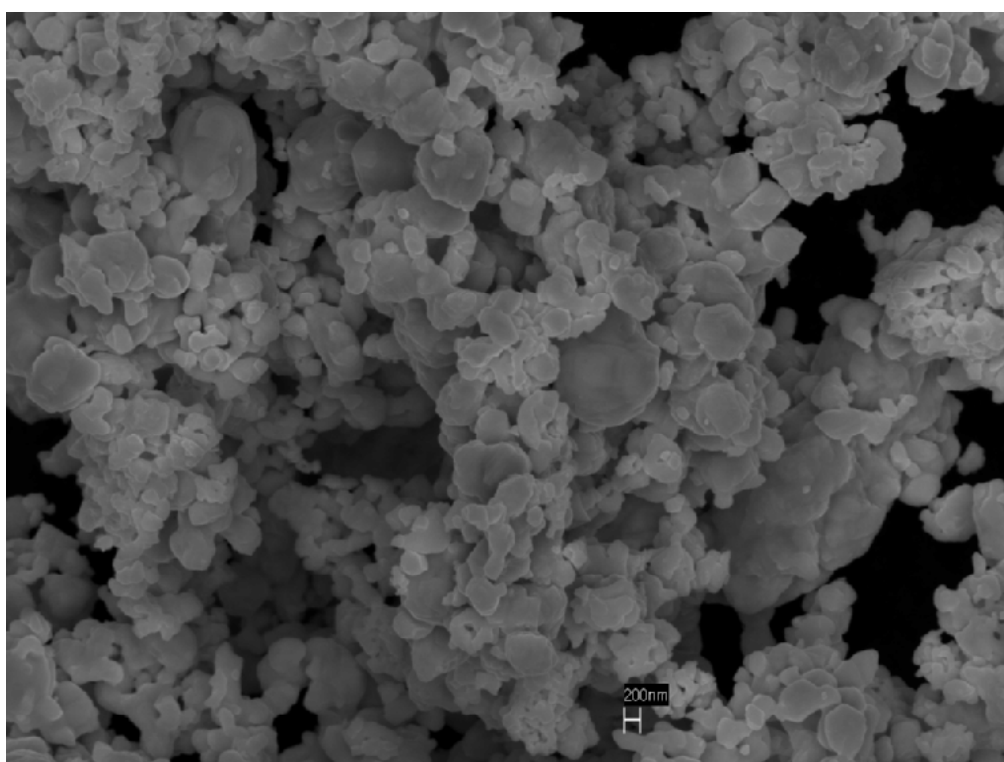


Fig B15 SHM008 - 30k nominal magnification

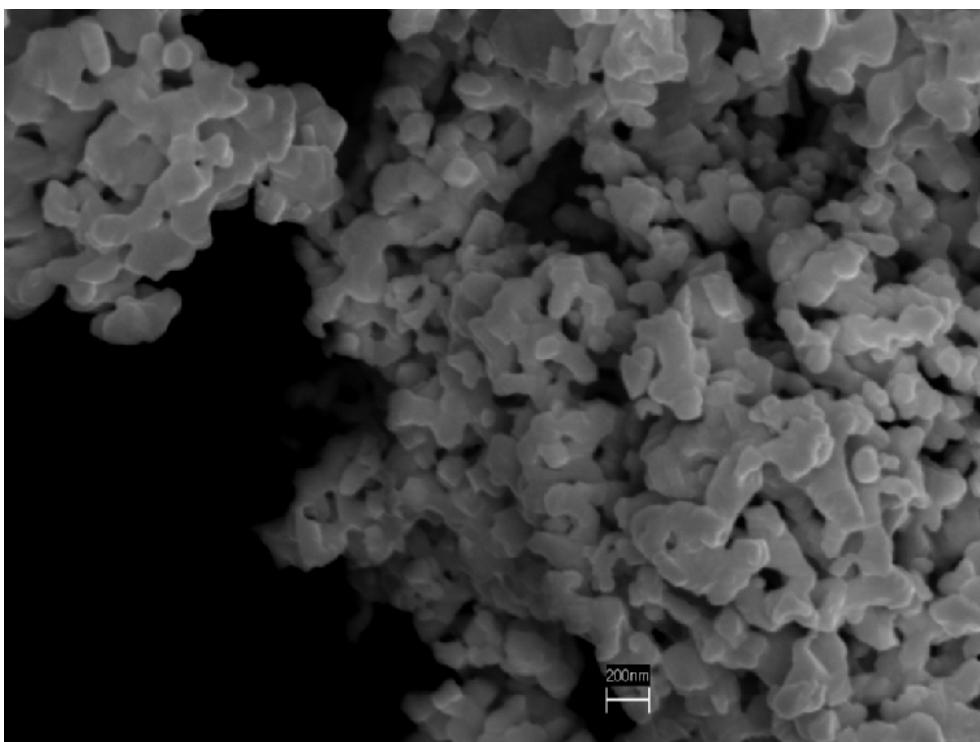


Fig B16 SHM002 - 80k nominal magnification

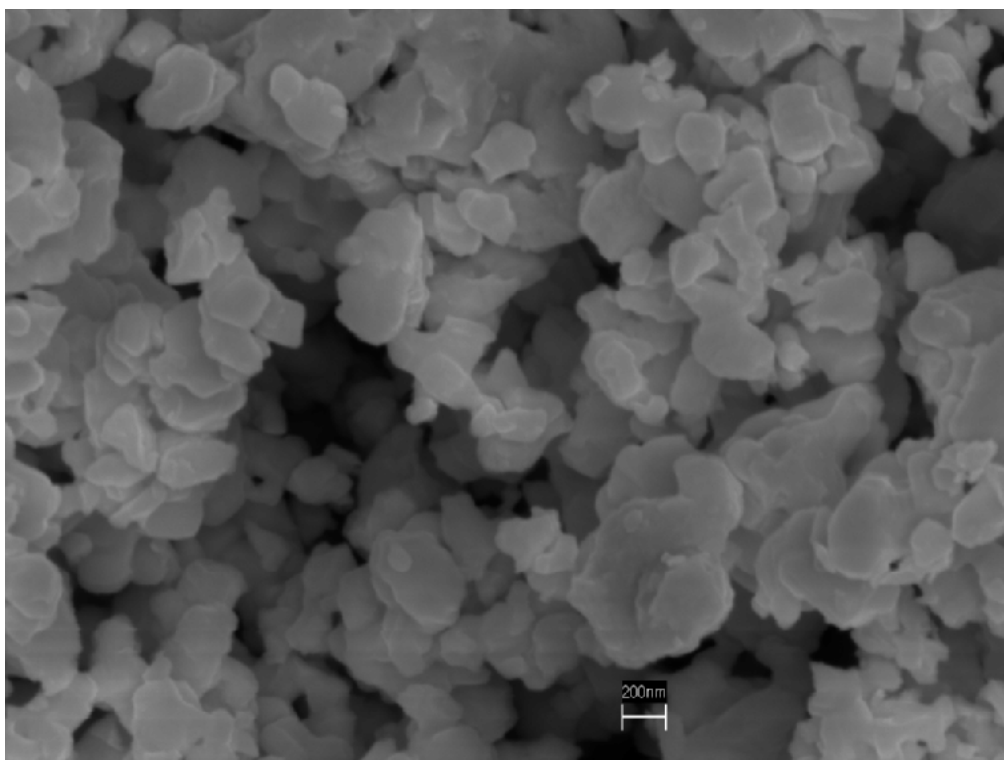


Fig B17 SHM004 - 80k nominal magnification

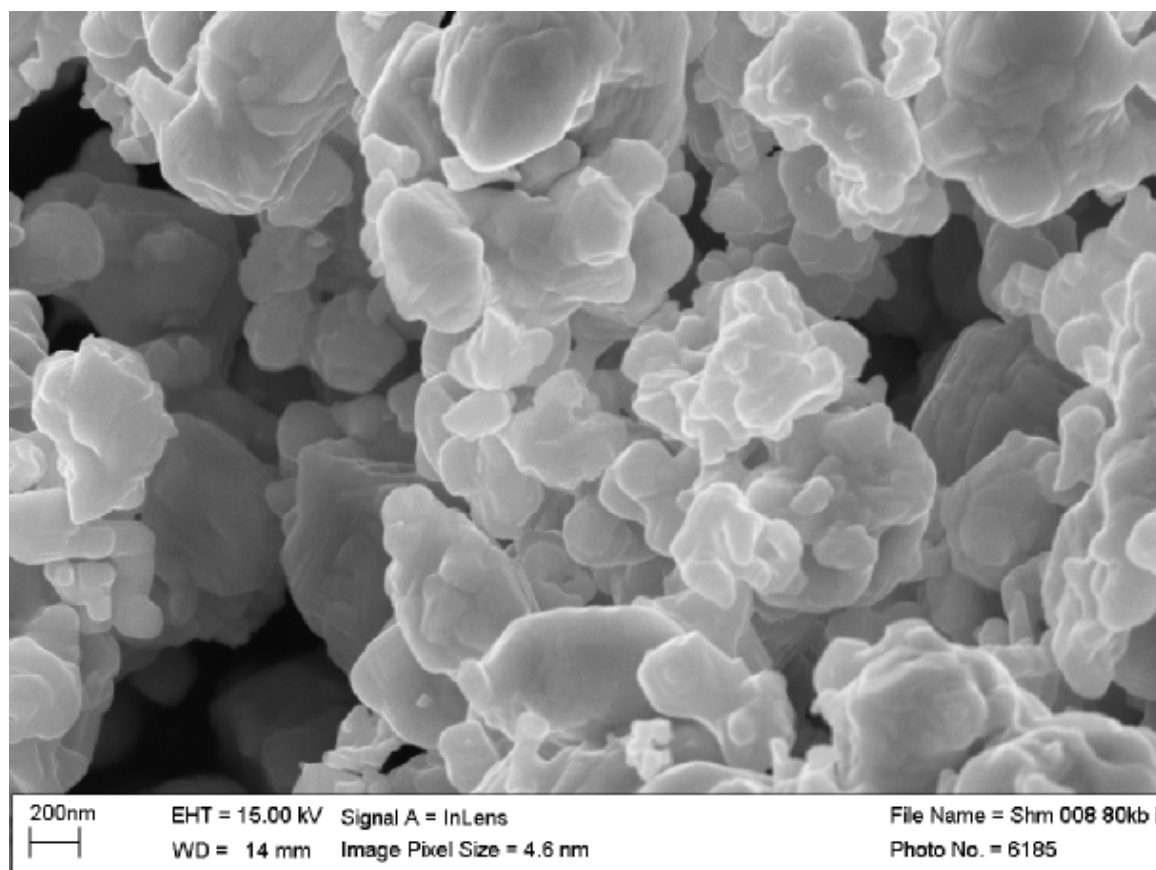


Fig B18 SHM008 - 80k nominal magnification

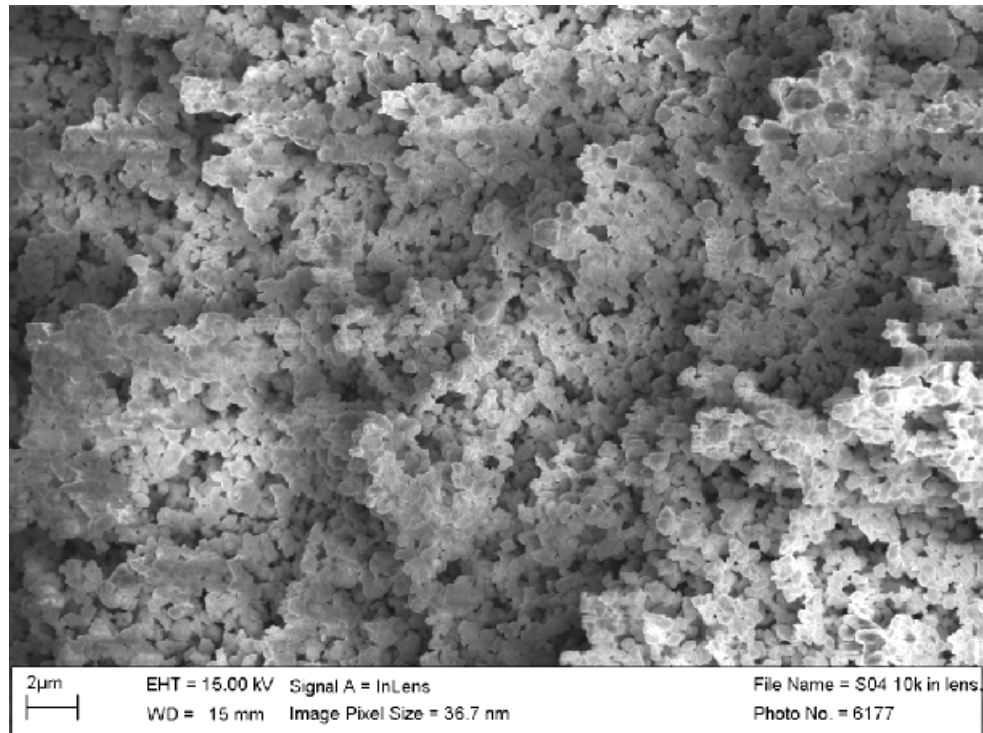


Fig B19 SO4C - 10k nominal magnification

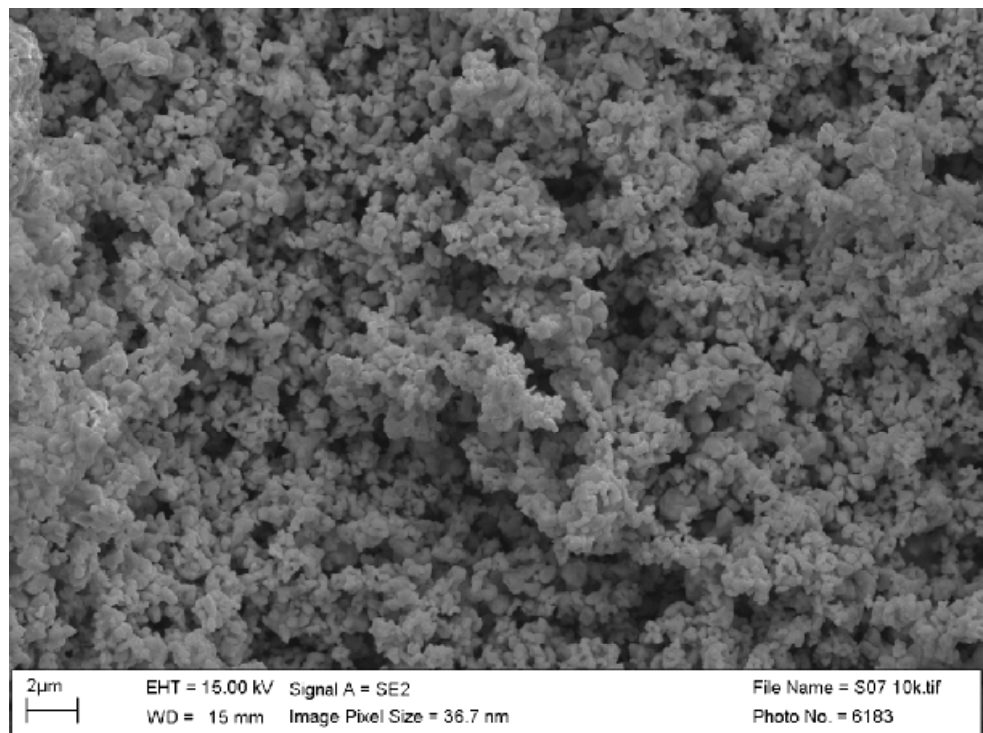


Fig B20 SO7V - 10k nominal magnification

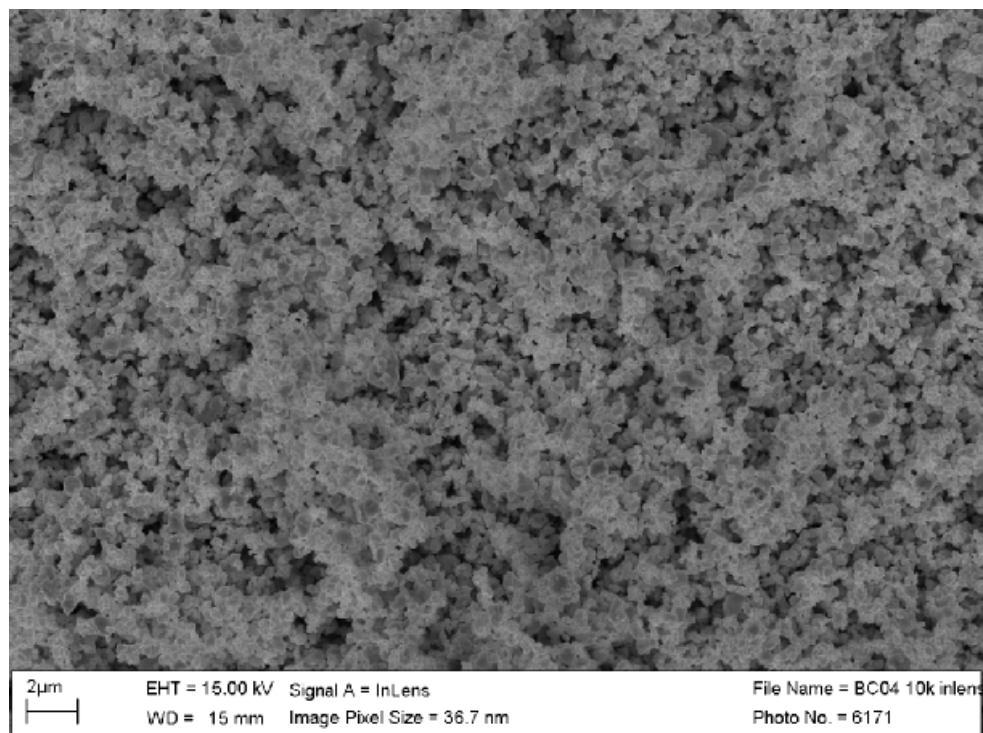


Fig B21 BCO4 - 10k nominal magnification

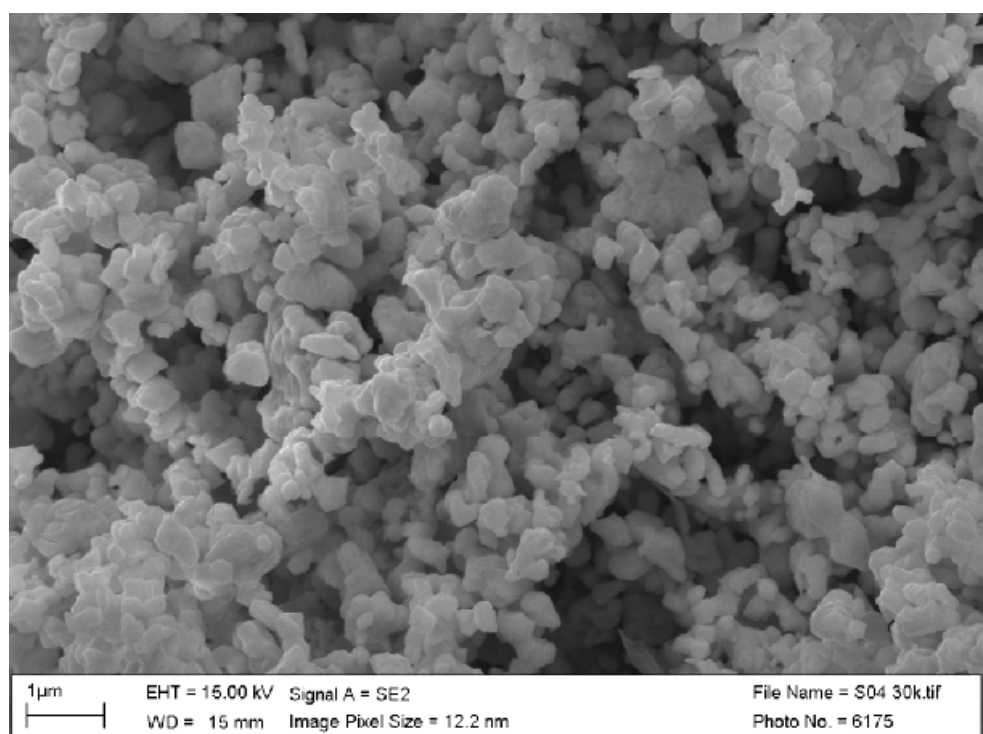


Fig B22 SO4C - 30k nominal magnification

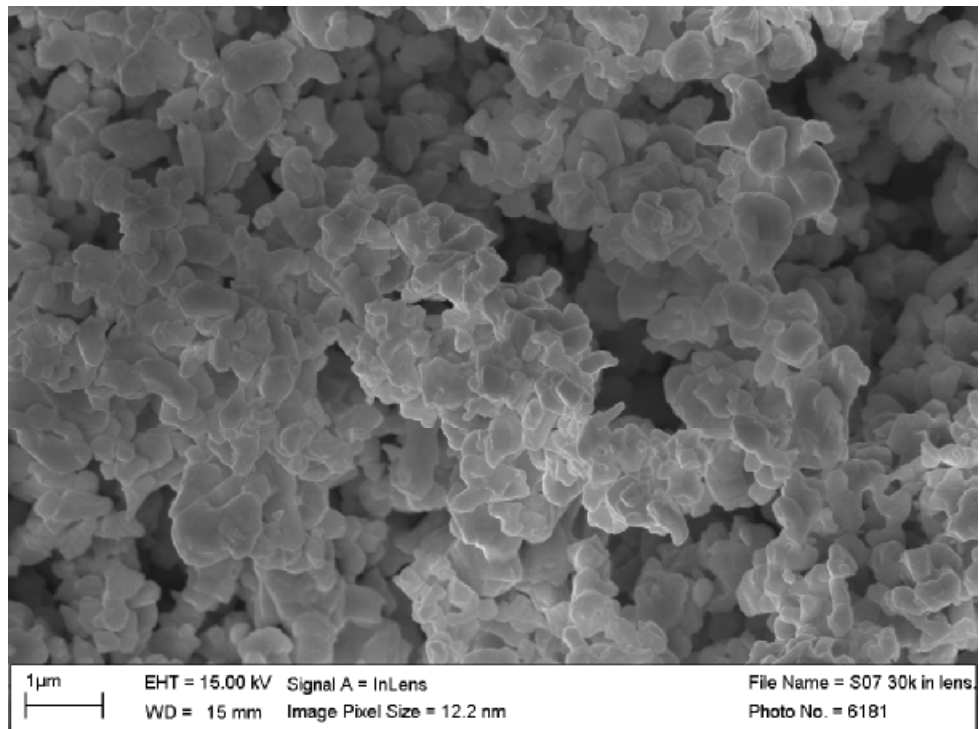


Fig B23 SO7V - 30k nominal magnification

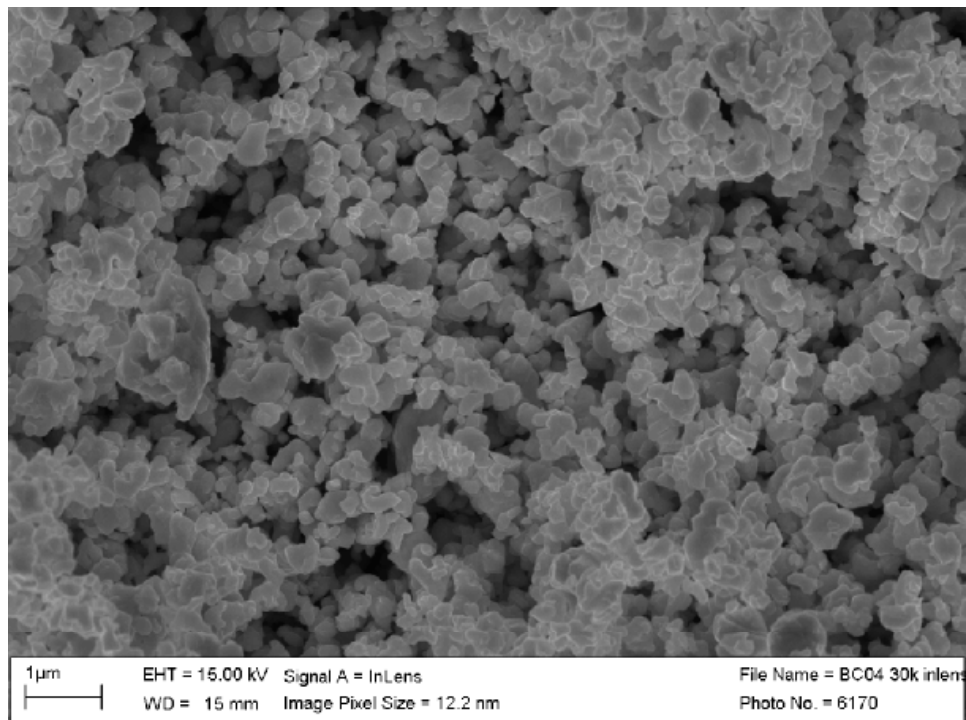


Fig B24 BCO4 - 30k nominal magnification

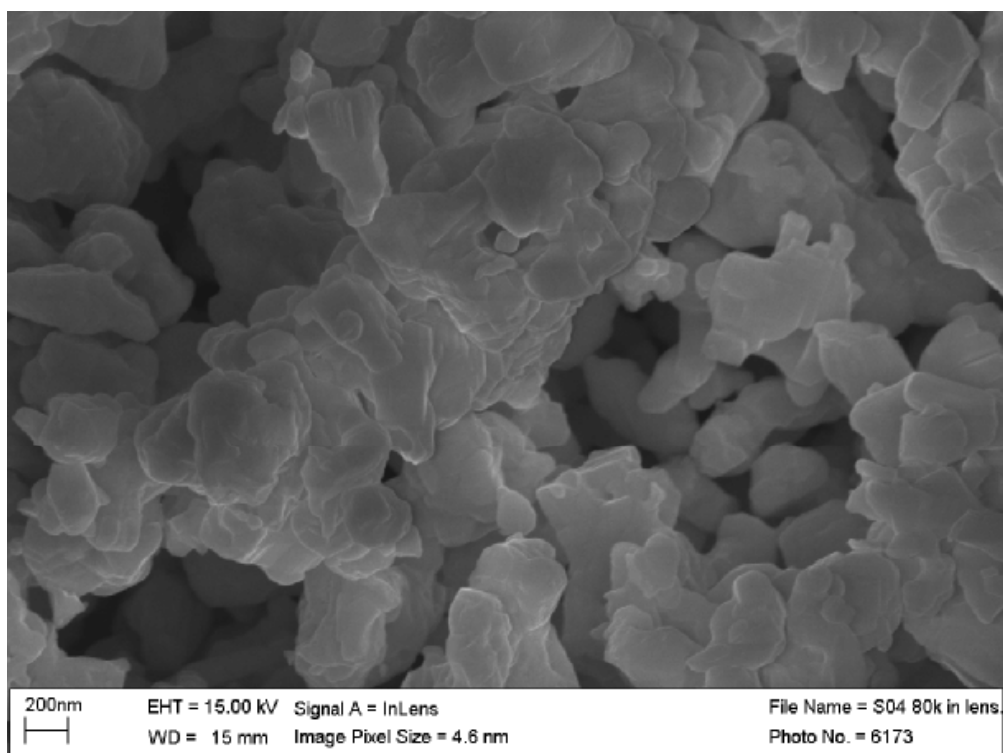


Fig B25 SO4C - 80k nominal magnification

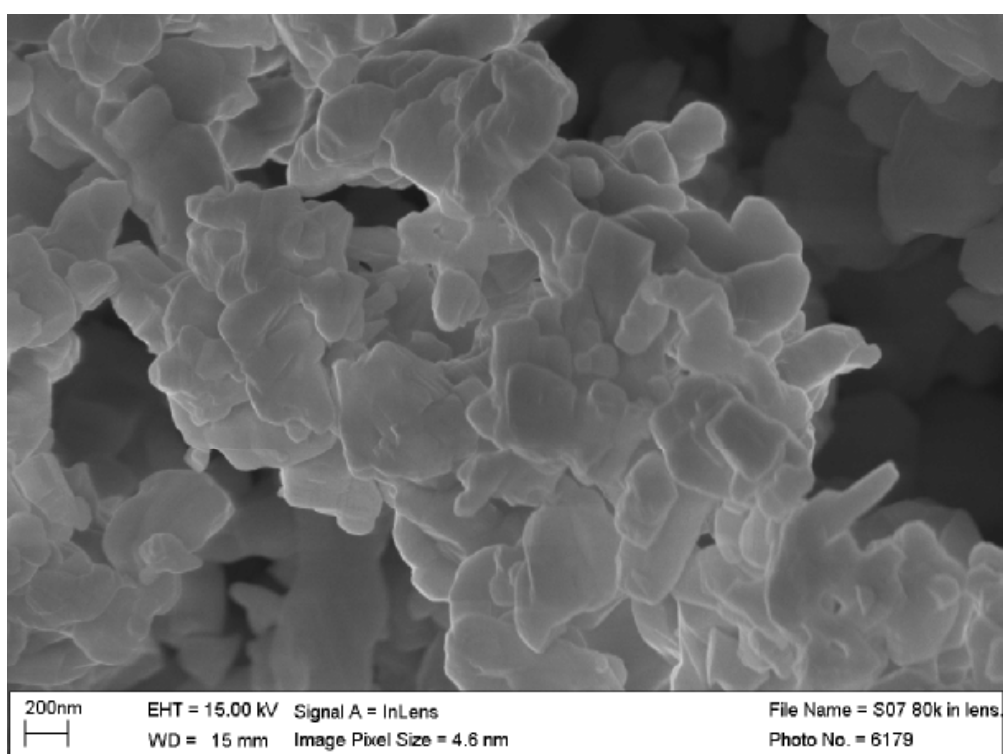


Fig B26 SO7V - 80k nominal magnification

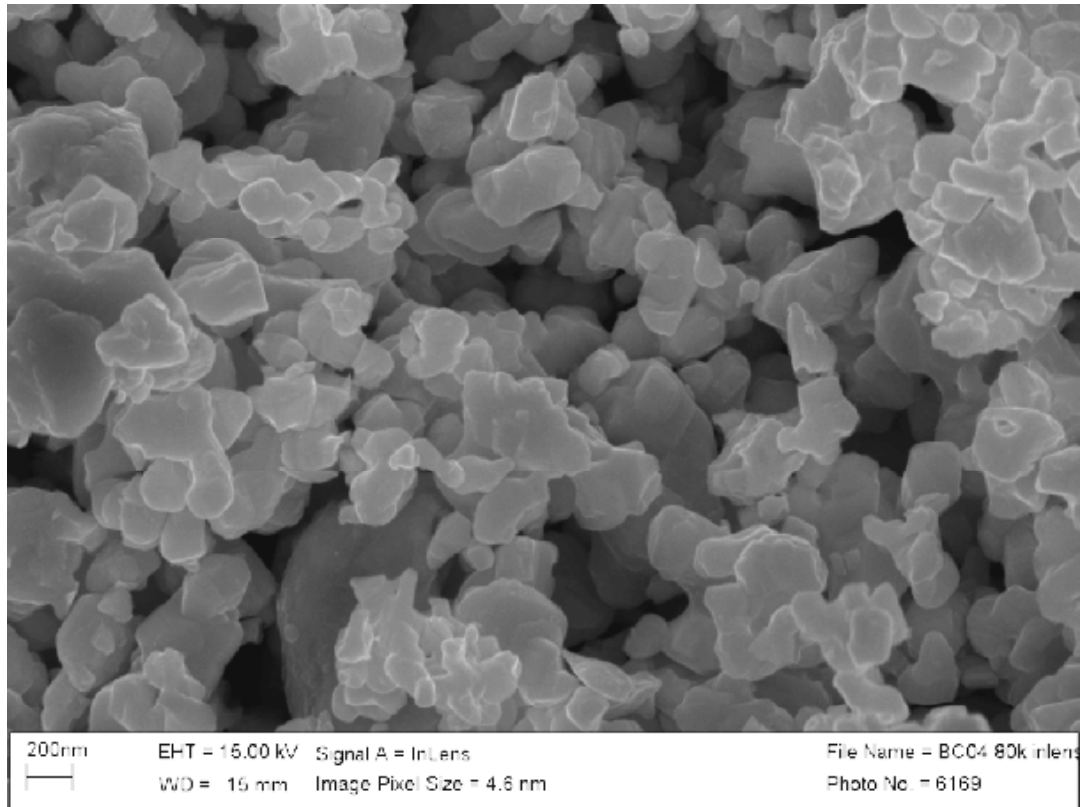


Fig B27 BCO4 - 80k nominal magnification

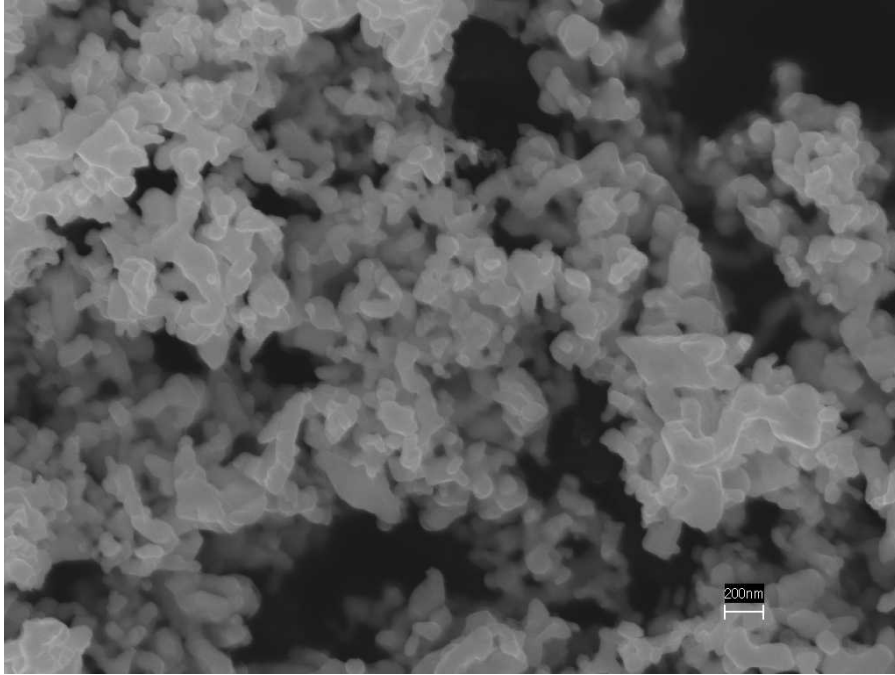


Fig B28 ALMT Corp. WC powder - 80k nominal magnification

APPENDIX C
EBSD MAPS OF POLISHED SECTIONS OF
POWDERS MOUNTED IN SILVER

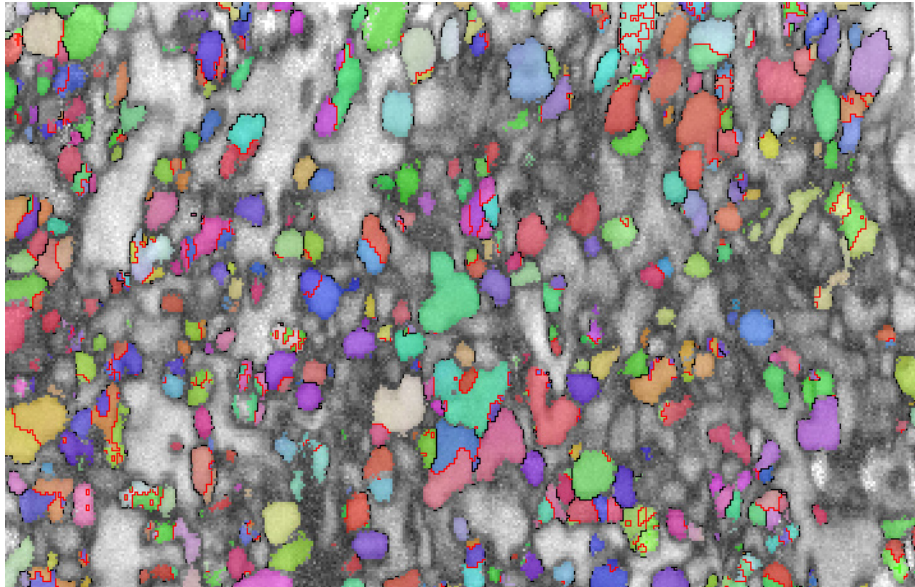


Fig D1 EBSD map of WBH powder (CRC15).

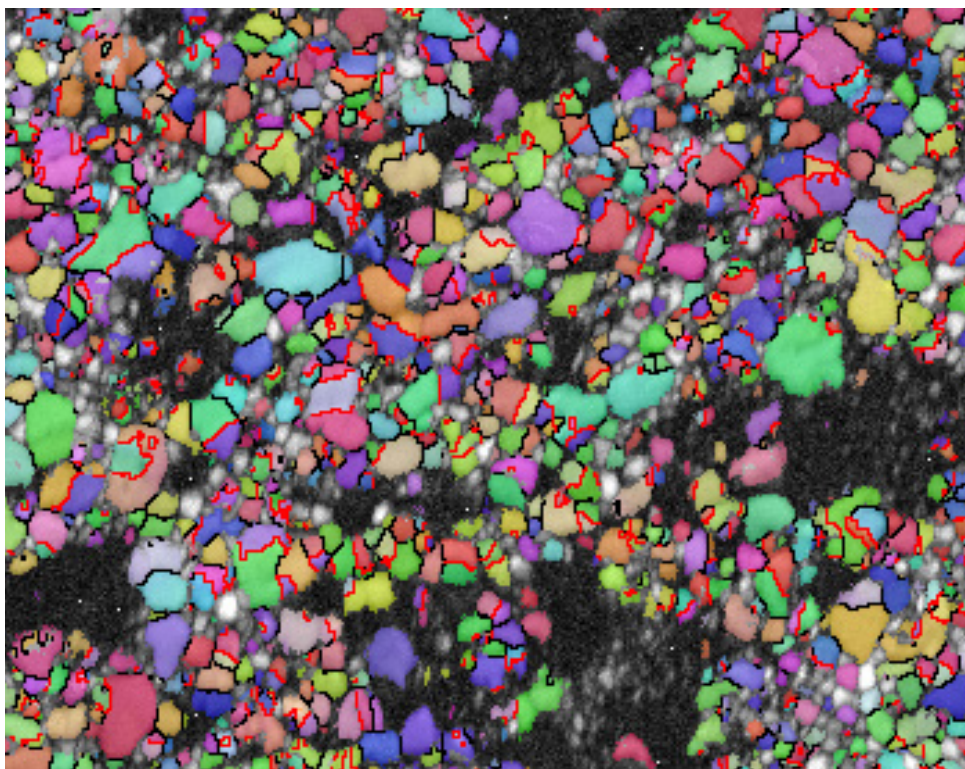


Fig D2 EBSD map of WBH powder (CRC025).

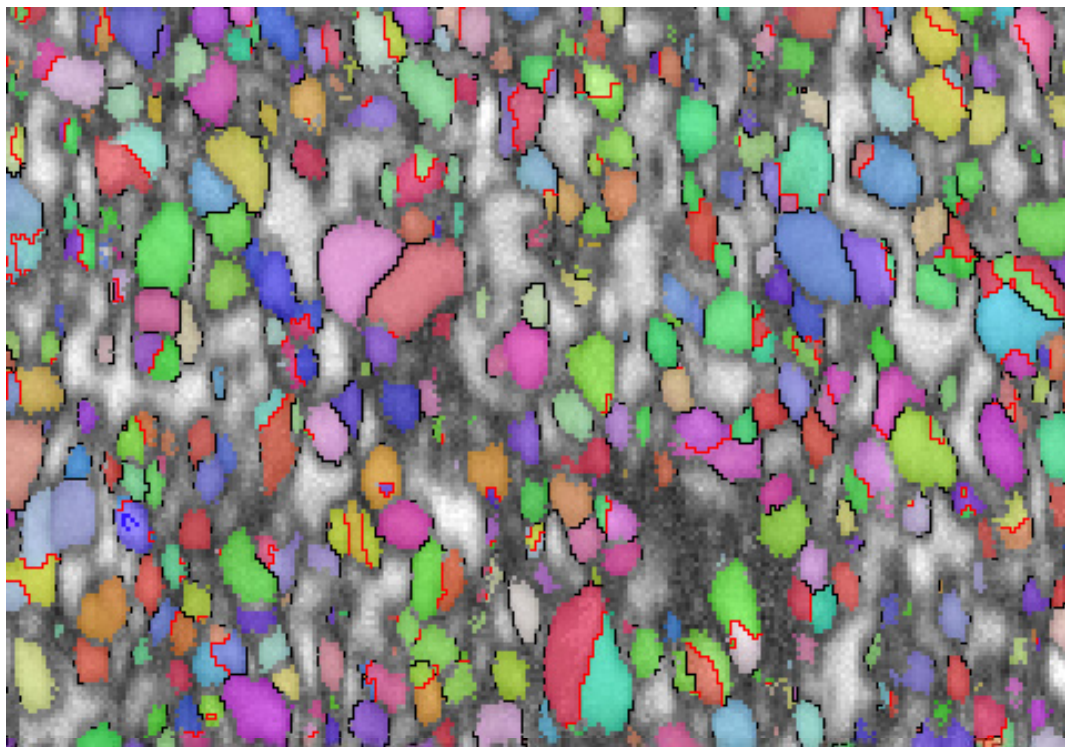


Fig D3 EBSD map of WBH powder (CRC30-40).

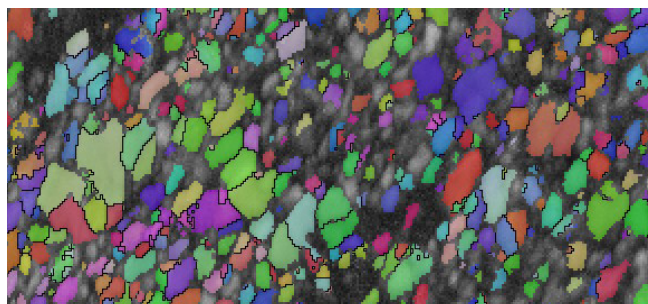


Fig D4 EBSD map of SHM powder (SHM002).

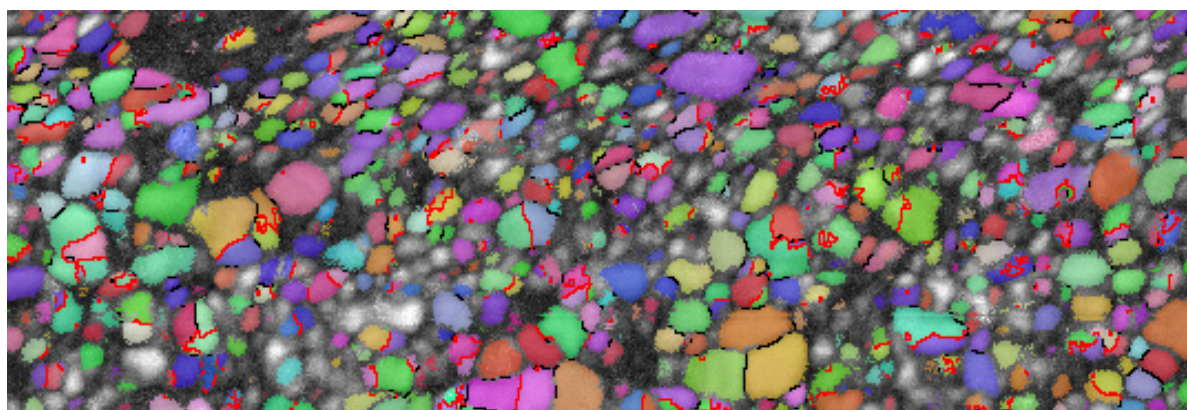


Fig D5 EBSD map of SHM powder (SHM004).

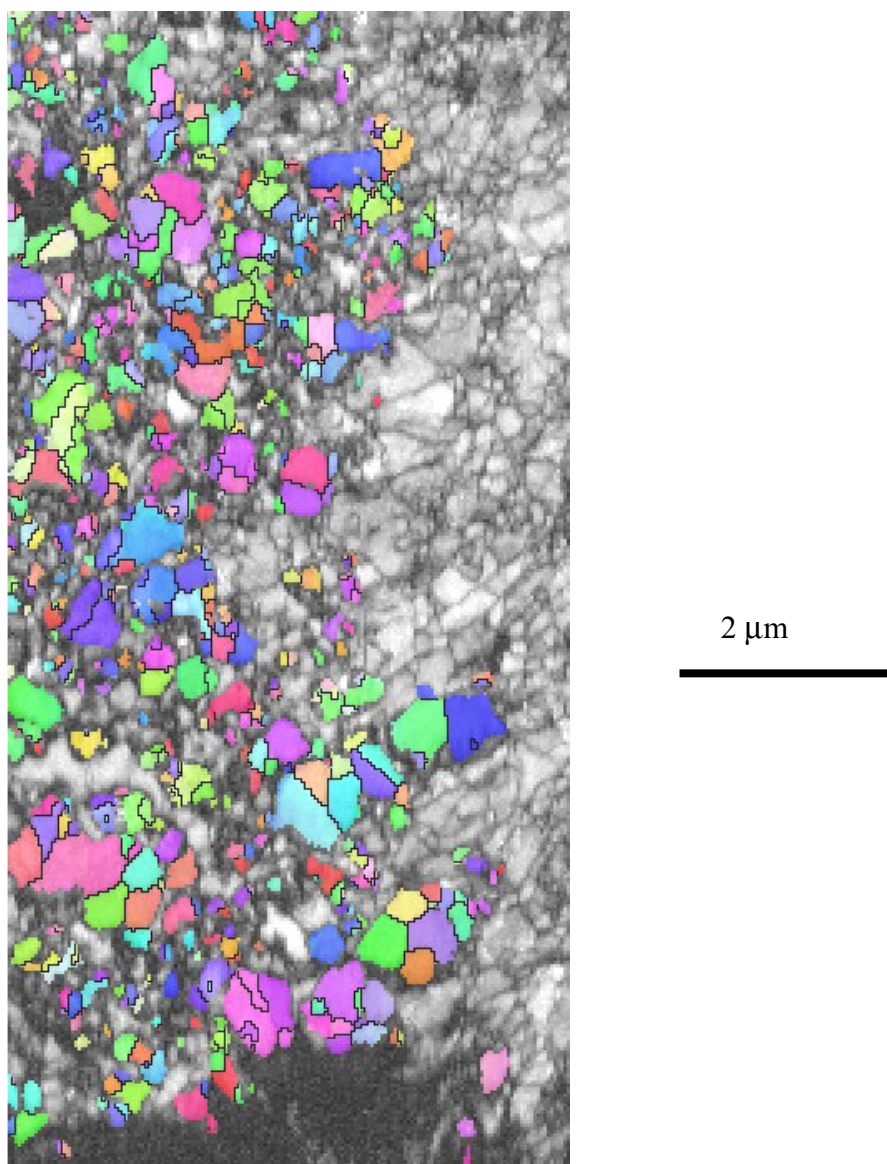


Fig D6 EBSD map of SHM powder (SHM008).

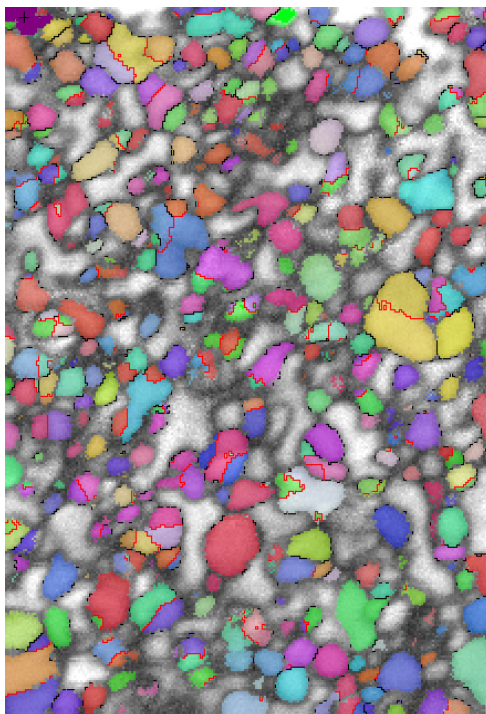
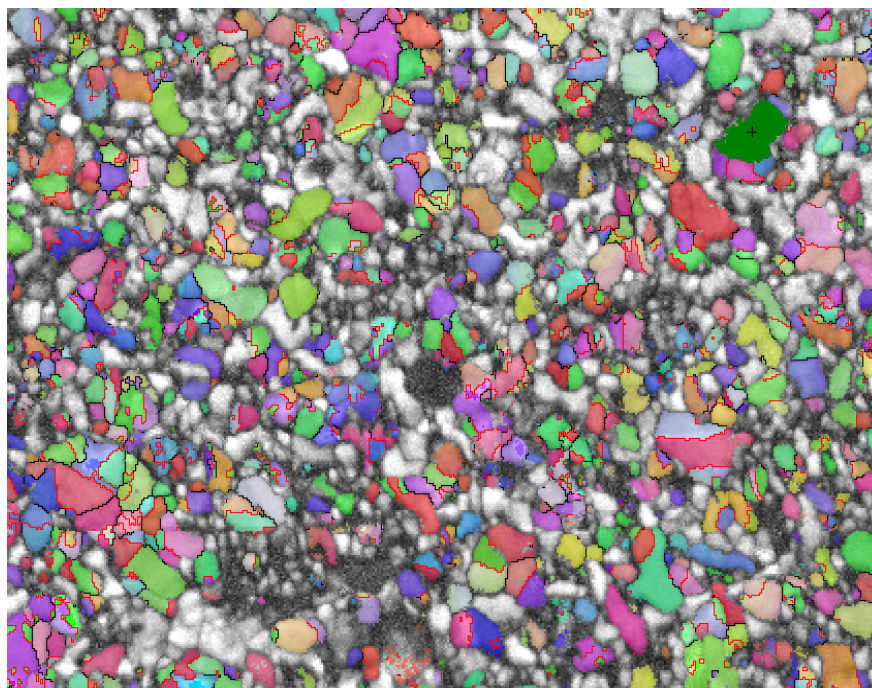
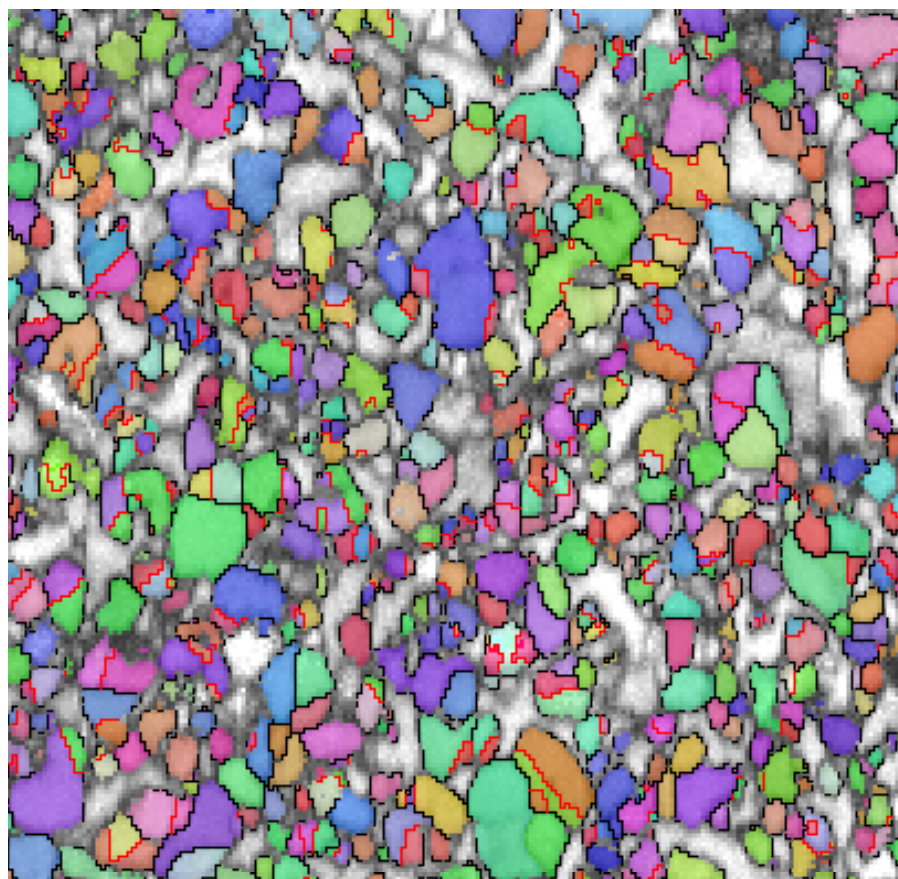


Fig D7 EBSD map of GTP powder (BC04).



2 μm

Fig D8 EBSD map of GTP powder (S04C).



2 μm

Fig D9 EBSD map of GTP powder (S07V).

APPENDIX D

INTERLABORATORY DATA

HM Analysis

Table D1 – WBH data

Source data - WBH	WBH powders			SHM powders			GTP (Osrarn Sylvania) powders		
	1	2	3	4	5	6	7	8	9
Laboratory Code	754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG	QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG
	CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002	SO7V	SO4C	BC04
BET specific surface area [m ² /g]	1.31	1.70	2.42	1.32	1.73	2.13	1.12	1.39	1.89
Calculated BET size (BET) [µm]	0.29	0.22	0.16	0.29	0.22	0.18	0.34	0.27	0.20
FSSS number [µm] as supplied	0.80	0.65	0.51	0.99	0.65	0.61	0.83	0.72	0.62
FSSS number [µm] ASTM deagglomerated	0.83	0.69	0.57	0.86	0.70	0.57	0.84	0.73	0.67
CILAS [µm] (median size)	0.73	0.65	0.48	1.05	0.65	0.68	0.89	0.72	0.57
Pressing density [g/cm ³]	7.85	7.46	6.98	6.59	7.53	5.98	7.14	7.33	7.56
Porosity (as supplied)	0.625	0.665	0.690	0.696	0.679	0.761	0.695	0.676	0.665
Porosity (ASTM) deagglomerated	0.598	0.621	0.660	0.613	0.630	0.666	0.630	0.635	0.631
Al [ppm]	<1	1	1	2	<1	3	2	<1	<1
As [ppm]	<20	<20	<20	<20	<20	<20	<20	<20	<20
Free carbon [%]	0.03	0.04	0.04	0.04	0.02	0.08	0.05	0.12	0.01
Total carbon [%]	6.13	6.12	6.08	6.10	6.03	6.06	6.10	6.09	6.07
Ca [ppm]	3	<2	3	11	7	6	16	6	2
Cd [ppm]	<2	<2	<2	<2	<2	<2	<2	<2	<2
Co [ppm]	<5	<5	<5	7	7	19	14	15	6
Cr [ppm]	18	14	42	12	9	5	44	#	22
Cu [ppm]	<2	<2	<2	<2	<2	<2	3	3	<2
Fe [ppm]	36	28	39	56	110	19	88	63	120
K [ppm]	<4	<4	<4	6	4	7	16	<4	<4
Li [ppm]	<2	<2	<2	<2	<2	<2	<2	<2	<2
Mg [ppm]	<1	<1	<1	<1	<1	1	<1	<1	<1

Source data - WBH		WBH powders			SHM powders				GTP (Osram Sylvania) powders			
		1	2	3	4	5	6	7	8	9		
Laboratory Code		754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG	QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG		
		CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002	SO7V	SO4C	BC04		
	Mn [ppm]	< 2	< 2	< 2	< 2	< 2	< 2	2	2	3		
	Mo [ppm]	10	< 10	< 10	11	< 10	13	11	15	< 10		
	Na [ppm]	15	14	18	5	4	8	3	16	4		
	Nb [ppm]	< 5	< 5	< 5	< 5	< 5	< 5	31	29	< 5		
	Ni [ppm]	< 5	< 5	< 5	7	< 5	< 5	31	35	10		
	O [ppm]	990	1280	1850	1920	2210	3170	2000	3230	1770		
	N [ppm]											
	P [ppm]	< 20	< 20	< 10	< 20	< 20	< 20	< 20	< 20	< 20		
	Pb [ppm]	< 10	< 10	< 2	< 10	< 10	< 10	< 10	< 10	< 10		
	S [ppm]	< 5	< 5	< 5	5	< 5	< 5	< 5	< 5	< 5		
	Si [ppm]	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10		
	Sn [ppm]	< 10	< 10	< 2	< 10	< 10	< 10	< 10	< 10	< 10		
	Ta [ppm]	< 5	< 5	< 5	< 5	< 5	< 5	39	41	< 5		
	Ti [ppm]	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5		
	V [ppm]	9	6	10	< 5	< 5	< 5	#	#	< 5		
	VC-anal. [%]	#	#	#	#	#	#	0.29	0.39	#		
	Cr ₃ C ₂ -anal. [%]	#	#	#	#	#	#	#	0.49	#		

Table D1 continued – WBH data

Table D2 – SHM data

Source data - SHM	WBH powders			SHM powders			GTP (Osram Sylvania) powders		
	1	2	3	4	5	6	7	8	9
Laboratory Code	754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG	QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG
	CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002	SO7V	SO4C	BC04
BET specific surface area [m ² /g]	1.23	1.59	2.2	1.2	1.90	2.40	1.04	1.30	1.80
Calculated BET size (BET) [μm]	0.31	0.24	0.17	0.32	0.20	0.16	0.37	0.29	0.21
FSSS number [μm] as supplied	0.65	0.55	0.55	0.95	0.75	0.56	0.81	0.71	0.60
FSSS number [μm] ASTM deagglomerated	0.65	0.55	0.55	0.84	0.66	0.55	0.80	0.65	0.62
CILAS [μm] (median size)									
Pressing density [g/cm ³]									
Porosity (as supplied)				0.72	0.74	0.78	0.72		
Porosity (ASTM) deagglomerated									
Al [ppm]									
As [ppm]									
Free carbon [%]	< 0.020	0.030	0.029	0.030	0.030	0.010	< 0.020	0.030	< 0.020
Total carbon [%]	6.13	6.12	6.08	6.10	6.08	6.07	6.10	6.10	6.07
Ca [ppm]									
Cd [ppm]									
Co [ppm]									
Cr [ppm]	300	100	100				< 500	4300	< 500
Cu [ppm]									
Fe [ppm]									
K [ppm]									
Li [ppm]									
Mg [ppm]									
Mn [ppm]									
Mo [ppm]									

Source data - SHM		WBH powders			SHM powders				GTP (Osram Sylvania) powders			
		1	2	3	4	5	6		7	8	9	
Laboratory Code		754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG		QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG	
		CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002		SO7V	SO4C	BC04	
Na [ppm]												
Nb [ppm]												
Ni [ppm]												
O [ppm]		1600	2300	2700					2500	3900	2400	
N[ppm]		200	240	330					400	600	600	
P [ppm]												
Pb [ppm]												
N [ppm]												
S [ppm]												
Si [ppm]												
Sn [ppm]												
Ta [ppm]												
Ti [ppm]												
V [ppm]		200	100	0					2400	3300	< 100	
VC-anal. [%]												
Cr ₃ C ₂ -anal. [%]												

Table D2 continued – SHM data

Table D3 – GTP data (mean values; full data set in Table 6)

Source data - GTP		WBH powders			SHM powders			GTP (Osram Sylvania) powders		
	1	2	3	4	5	6	7	8	9	
Laboratory Code	754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG	QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG	
	CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002	SO7V	SO4C	BC04	
BET specific surface area [m ² /g]	1.31	1.49	1.83	1.25	1.73	2.13	1.13	1.43	1.53	
Calculated BET size (BET) [μm]	0.29	0.26	0.21	0.30	0.22	0.18	0.34	0.27	0.25	
FSSS number [μm] as supplied	0.77	0.64	0.52	0.94	0.61	0.56	0.79	0.66	0.58	
FSSS number [μm] Rod milled	0.78	0.64	0.52	0.88	0.70	0.56	0.74	0.69	0.68	
CILAS [μm] (median size)										
Pressing density [g/cm ³]										
Porosity (as supplied)	0.62	0.66	0.69	0.69	0.66	0.75	0.67	0.65	0.65	
Porosity (ASTM) deagglomerated	0.59	0.62	0.64	0.59	0.61	0.66	0.62	0.62	0.63	
Al [ppm]										
As [ppm]										
Free carbon [%]										
Total carbon [%]										
Ca [ppm]										
Cd [ppm]										
Co [ppm]										
Cr [ppm]										
Cu [ppm]										
Fe [ppm]										
K [ppm]										
Li [ppm]										
Mg [ppm]										
Mn [ppm]										
Mo [ppm]										

Source data - GTP	WBH powders			SHM powders			GTP (Osram Sylvania) powders		
	1	2	3	4	5	6	7	8	9
Laboratory Code	754/05/WCV	336/M/05/X WCV	376/M/06/W CV	QWC/NPL/ 7935-M2554- 12/AWG	QWC/NPL/ 7929-M2554- 11/AWG	QWC/NPL/ 7919-M2554- 7/AWG	QWC/NPL/ 7950-M2554- 3/AWG	QWC/NPL/ 7957-M2554- 5/AWG	QWC/NPL/ 7939-M2554- 1/AWG
	CRC030-40	CRC025U	CRC015U	SHM008	SHM004	SHM002	SO7V	SO4C	BC04
Na [ppm]									
Nb [ppm]									
Ni [ppm]									
O [ppm]									
P [ppm]									
Pb [ppm]									
S [ppm]									
Si [ppm]									
Sn [ppm]									
Ta [ppm]									
Ti [ppm]									
V [ppm]									
VC-anal. [%]									
Cr ₃ C ₂ -anal. [%]									

Table D3 continued – GTP data

Table D4 – GTP data, full set

LIMS #	Company	Sample	Grade	BET		Rod Mill BET		FSSS		Rod Mill FSSS		XRD	RM XRD	MT3500			Rod Mill MT3500			MTX100			Rod Mill MTX100		
				Dia.	Surface area	Dia.	Surface area	size	porosity	size	porosity	Xtal size	Xtal size	10%	50%	90%	100	500	900	10%	50%	90%	100	500	900
80040067	Wolfram Bergbau	7906	CRC015U	0.22	1.78	0.16	2.33	0.52	0.685	0.52	0.64	212	321	0.184	0.453	0.984	0.161	0.379	0.816	0.37	0.63	1.05	0.34	0.57	0.93
80040068	Wolfram Bergbau	7902	CRC015U	0.20	1.88	0.16	2.42	0.52	0.69	0.52	0.64	324	316	0.175	0.448	1.077	0.155	0.377	0.841	0.37	0.63	1.04	0.34	0.57	0.94
80040069	Wolfram Bergbau	7907	CRC025U	0.30	1.27	0.23	1.68	0.64	0.655	0.64	0.615	363	358	0.199	0.579	1.231	0.176	0.494	1.034	0.45	0.76	1.13	0.39	0.70	1.07
80040070	Wolfram Bergbau	7908	CRC025U	0.23	1.70	0.21	1.81	0.64	0.655	0.64	0.615	370	366	0.202	0.574	1.227	0.170	0.480	1.011	0.45	0.77	1.13	0.39	0.70	1.06
80040071	Wolfram Bergbau	7915	CRC030-40	0.29	1.33	0.28	1.38	0.78	0.62	0.78	0.58	353	356	0.235	0.682	1.362	0.194	0.608	1.217	0.54	0.85	1.23	0.48	0.81	1.18
80040072	Wolfram Bergbau	7917	CRC030-40	0.30	1.29	0.29	1.34	0.76	0.625	0.78	0.585	363	352	0.222	0.657	1.355	0.189	0.603	1.198	0.52	0.85	1.21	0.48	0.81	1.17
80040325	Sandvik Hard Materials	7922	SHM002	0.17	2.22	0.15	2.53	0.56	0.745	0.56	0.665	353	327	0.378	1.531	9.65	0.153	0.421	2.494	0.58	2.08	10.48	0.34	0.68	1.72
80040326	Sandvik Hard Materials	7924	SHM002	0.19	2.03	0.16	2.33	0.56	0.745	0.56	0.655	378	351	0.332	1.417	10.66	0.154	0.415	2.215	0.57	1.77	9.23	0.34	0.70	2.46
80040327	Sandvik Hard Materials	7925	SHM004	0.22	1.71	0.21	1.82	0.6	0.665	0.7	0.61	311	303	0.185	0.526	1.132	0.164	0.450	1.002	0.42	0.73	1.09	0.36	0.69	1.07
80040328	Sandvik Hard Materials	7926	SHM004	0.22	1.74	0.20	1.89	0.62	0.66	0.7	0.615	339	311	0.19	0.538	1.134	0.162	0.449	1.031	0.42	0.73	1.1	0.37	0.69	1.06
80040329	Sandvik Hard Materials	7936	SHM008	0.31	1.24	0.26	1.50	0.94	0.685	0.88	0.59	383	367	0.501	1.912	7.62	0.195	0.703	2.864	0.8	2.04	7.36	0.47	1.05	2.69
80040330	Sandvik Hard Materials	7931	SHM008	0.31	1.26	0.25	1.53	0.94	0.685	0.88	0.595	391	348	0.521	1.962	7.6	0.201	0.738	2.875	0.84	2.09	7.22	0.49	1.07	2.81
80046610	Towanda	7961	S04CV	0.27	1.44	0.26	1.45	0.66	0.650	0.70	0.620	227	262	0.2230	0.628	1.461	0.233	0.593	1.345	0.49	0.82	1.26	0.45	0.77	1.19
80046611	Towanda	7964	S04CV	0.27	1.41	0.25	1.52	0.66	0.650	0.68	0.620	239	252	0.2260	0.629	1.488	0.218	0.577	1.329	0.48	0.82	1.26	0.44	0.77	1.18
80046612	Towanda	7954	S07V2	0.34	1.11	0.31	1.24	0.78	0.665	0.68	0.615	462	471	0.442	0.990	3.08	0.300	0.720	2.092	0.65	1.73	2.85	0.50	0.93	1.69
80046613	Towanda	7955	S07V2	0.33	1.15	0.31	1.22	0.80	0.665	0.80	0.615	471	501	0.447	0.986	3.02	0.300	0.715	2.088	0.65	1.13	2.79	0.50	0.92	1.72
80040537	Bruntal	----	BC04U	0.25	1.53	0.20	1.89	0.58	0.65	0.68	0.63	201	304	0.183	0.48	0.912	0.160	0.428	0.872	0.4	0.65	0.95	0.37	0.63	0.94

APPENDIX E

HEL FSSS DATA

Table E1

WBH Powders			
NPL Code	Supplier Code	Porosity (as-supplied)	Particle size μm
7901	CRC015	0.7075	0.51
7901	CRC015	0.6575	0.60
7907	CRC025	0.7075	0.66
7907	CRC025	0.6575	0.67
7913	CRC030-40	0.7075	0.93
7913	CRC030-40	0.6575	0.88
7913	CRC030-40	0.6075	0.92

SHM Powders			
NPL Code	Supplier Code	Porosity (as-supplied)	Particle size μm
7923	SHM002	0.8085	0.751
7923	SHM002	0.7585	0.63
7923	SHM002	0.7329	0.63
7925	SHM004	0.7075	0.74
7925	SHM004	0.6575	0.72
7934	SHM008	0.7578	1.239
7934	SHM008	0.7328	1.152
7934	SHM008	0.6828	1.09

GTP Powders			
NPL Code	Supplier Code	Porosity (as-supplied)	Particle size μm
7937	BC04	0.7577	1.10
7937	BC04	0.7327	0.81
7937	BC04	0.7078	0.67
7937	BC04	0.6828	0.63
7937	BC04	0.6578	0.63
7937	BC04	0.6328	0.64
7958	SO4C	0.7072	0.76
7958	SO4C	0.6822	0.73
7958	SO4C	0.6572	0.74
7958	SO4C	0.6323	0.75
7947	S07V	0.7577	1.05
7947	S07V	0.7327	0.97
7947	S07V	0.7077	0.92
7947	S07V	0.6827	0.93
7947	S07V	0.6577	0.94

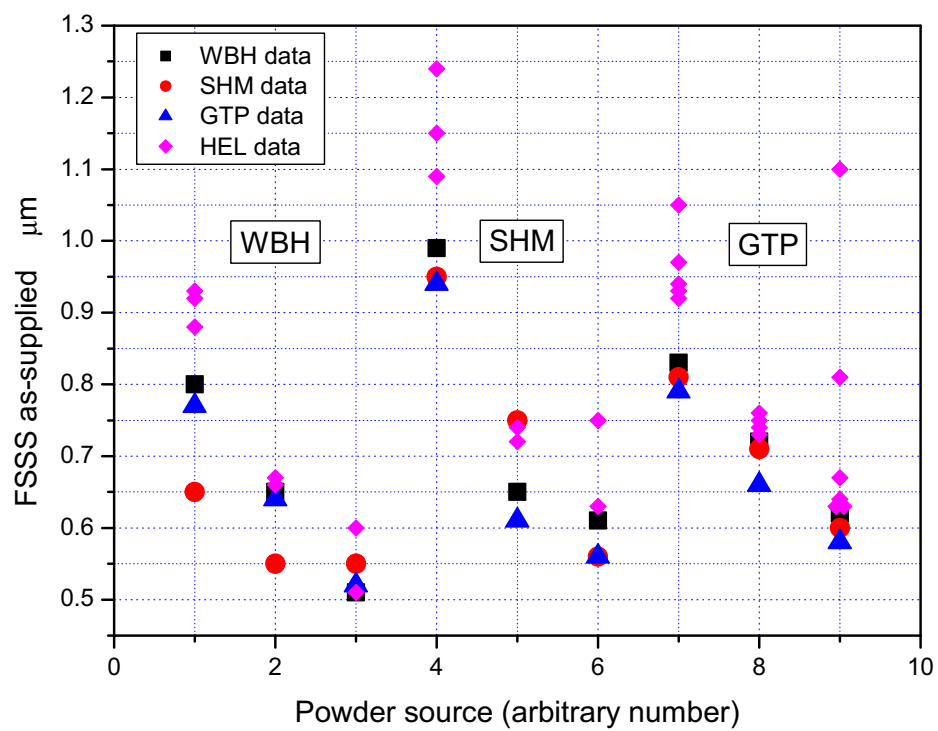


Fig E1 HEL FSSS data compared with Interlab results