National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1984

Thermal Spray Powder – Particle Size Distribution
Tungsten Carbide/Cobalt (Acicular)

This Standard Reference Material (SRM) is primarily intended for use in the calibration of equipment used to measure particle size distributions (PSD) in the 9 μm to 30 μm range. SRM 1984 consists of a single bottle containing approximately 14 g of tungsten carbide/cobalt powder.

The PSD values at five mass percentiles were measured by scanning electron microscopy (SEM) and laser light scattering (LLS). The certified PSD values by SEM are listed in Table 1. These certified values were determined by the measurement of over 10 000 individual particles from three bottles. The reference PSD values by LLS are listed in Table 2. A comparison of the two methods is shown in Figure 1.

Table 1. Certified PSD Values by SEM

Cumulative Mass Fraction (%)	Certified Diameter (μm)	Uncertainty[(a)] (μm)
10	10.3	0.9
25	13.2	0.9
50	17.1	2.2
75	21.3	1.6
90	26.3	0.9

[(a)] The uncertainty at each percentile, computed according to the ISO and NIST Guides [1], is an expanded uncertainty at the 95 % level of confidence.

Expiration of Certification: The certification of **SRM 1984** is valid until **31 August 2015** provided the sample dispersion procedures are rigorously followed (see "Sample Dispersion and Measurement Procedures for Light Scattering Method"). The powder contains no organic binder and is, therefore, considered chemically stable. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The concept for the development of this SRM was provided by S.J. Dapkunas of the NIST Ceramics Division.

The SRM measurement technique, development, and certification were performed by J.F. Kelly and P. Pei of the NIST Ceramics Division.

Statistical analyses were performed by H-k. Liu of the NIST Statistical Engineering Division.

Debra L. Kaiser, Chief
Ceramics Division

Gaithersburg, MD 20899
Certificate Issue Date: 07 November 2010
Certificate Revision History on Page 3.

Robert L. Watters, Jr., Chief
Measurement Services Division

Support aspects involved in the preparation and issuance of this SRM were coordinated through the NIST Measurement Services Division.

Measurement of SRM: The starting material was a 10 kg batch from a single lot (W2203A) of Type SD 251 tungsten carbide/cobalt powder obtained from OSRAM Sylvania Products, Inc.[1], Towanda, PA. This powder was chosen for its size distribution, irregularly shaped particle morphology (Figure 2), resistance to fracture on handling, and low level of aggregation.

The powder was split into bottle units containing approximately 14 g each by using spinning rifflers. A randomized set of 100 bottles from 672 bottles was selected for homogeneity testing, round robin study using LLS instruments, and certification analyses by SEM. Homogeneity testing of 14 randomly selected bottles, measured in duplicate, was performed with a LLS instrument at NIST. The data showed no evidence of size heterogeneity.

Scanning Electron Microscopy Analysis: SEM based image analysis was carried out on three bottles. Sample preparation for microscopy entailed both a reduction in the mass of powder and a separation into size fractions. The size fractionation was accomplished by sieving using sieves with nominal openings of 10 μm, 20 μm, and 30 μm. Subsamples from each of the sieve splits were then produced by successive division using a spinning riffler.

SEM images were acquired for each of the sieve fractions. The backscatter electron images of the particles were acquired as greyscale image files into a computer via a digital interface. The 2048 × 2048 pixel images were analyzed to obtain the projected areas of the tungsten carbide/cobalt particles. These areas were fitted with ellipses and the major and minor axes converted to particle volumes (prolate ellipsoids) and particle diameters using the average of the three ellipsoid axes. The pixel-to-length conversion was calibrated using a micrometer slide calibrated at NIST using laser interferometry. Several hundred particles of each sieve fraction were measured for a total of approximately 2 400 particles measured from each bottle. Particle size distributions describing the percent of powder mass represented by particles with diameters less than a given length were calculated using the weighting factors obtained from the sieving results. The diameter values corresponding to the specific mass fractions of 10 %, 25 %, 50 %, 75 %, and 90 % are listed in Table 1. Current practice in the thermal spray industry is to specify these values to define the particle size distribution. A graphical comparison of the distribution measured by SEM with the mean distribution obtained by LLS is shown in Figure 1.

Laser Light Scattering Method (Round Robin Study): Ten laboratories participated in this round robin study. Each round robin participant received two bottles for analysis using their light scattering instruments. The reference distribution values given in Table 2 are based on 20 measurements by the 10 participating laboratories. The LLS data from all 10 laboratories were obtained using such instruments as the Horiba LA910, Microtrac X100, Sympatec HELOS, and Beckman Coulter LS 230 following the sample preparation procedure specified by NIST. Therefore, in the use of this SRM, the sample dispersion procedure is mandatory; otherwise, the data may not be comparable. Although the light scattering instruments produce a continuous plot of weight percent finer than a given diameter, five cumulative percentiles were selected as a representative data set for certification since it is consistent with the industrial practice.

The following individuals and companies participated in the development of this SRM:

T. Weigel and P. Plantz, Honeywell, Microtrac Solution Center, Clearwater, FL
J. Wolfgang, SYMPATEC, Inc., Princeton, NJ
L. Vu, P. Fernando, and M. Pohl, Horiba Instruments, Inc., Irving, CA
L. Updegraff and R. Neiser, Sandia National Laborabories, Albuquerque, NM
J. Bohan, API, Hadley, MA
J. Vasiliou, Duke Scientific Corporation, Palo Alto, CA
K. Cowan and R. Iacocca, P/M Laboratory, Pennsylvania State University, State College, PA
H.D. Garrelts, Stellite Coatings, Goshen, IN
H. Hildebrand, Beckman Coulter, Miami, FL
R. Simmons, Dirats Laboratories, Westfield, MA
F. Venskytis, OSRAM Sylvania Products, Inc., Towanda, PA

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 2. PSD Reference Values by LLS

Cumulative Mass Fraction (%)	Reference Value (μm)	Uncertainty[(a)] (μm)
10	9.5	0.8
25	12.5	0.8
50	16.5	0.9
75	21.3	1.2
90	26.5	1.3

[(a)] The uncertainty at each percentile, computed according to the ISO and NIST Guides [1], is an expanded uncertainty at the 95 % level of confidence.

SAMPLE DISPERSION AND MEASUREMENT PROCEDURES FOR LIGHT SCATTERING METHOD

Sample Dispersion Procedure: Application of the reference values produced by LLS requires following this sample dispersion procedure; otherwise, results may not be comparable to the reference values listed in Table 2. **NOTE:** Each sample bottle contains approximately 14 g tungsten carbide/cobalt powder that is sufficient for many analyses by LLS instruments.

1. Use a microriffler for splitting the sample into subsamples of the appropriate mass as specified by the manufacturer's instructions.

2. Add 4 % aqueous sodium pyrophosphate solution to each subsample at the ratio of 1.00 mL/g of powder and make a paste by mixing gently with a spatula. **DO NOT use a magnetic stirrer since the powder will attach to the magnet and may be crushed during stirring.**

3. Transfer the paste quantitatively (totally) into the measuring cell containing distilled water whose pH has been pre-adjusted to 9.5 ± 0.1 with 1 mol/L sodium hydroxide solution. Flush the container with pH-adjusted distilled water to complete the transfer.

4. Follow the instrument manufacturers' instructions for instrument calibration and operation.

REFERENCE

[1] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2010); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/physlab/pubs/index.cfm (accessed Nov 2010).

Certificate Revision History: 07 November 2010 (Extension of the certification period; editorial changes); 01 December 2000 (Original certification date).

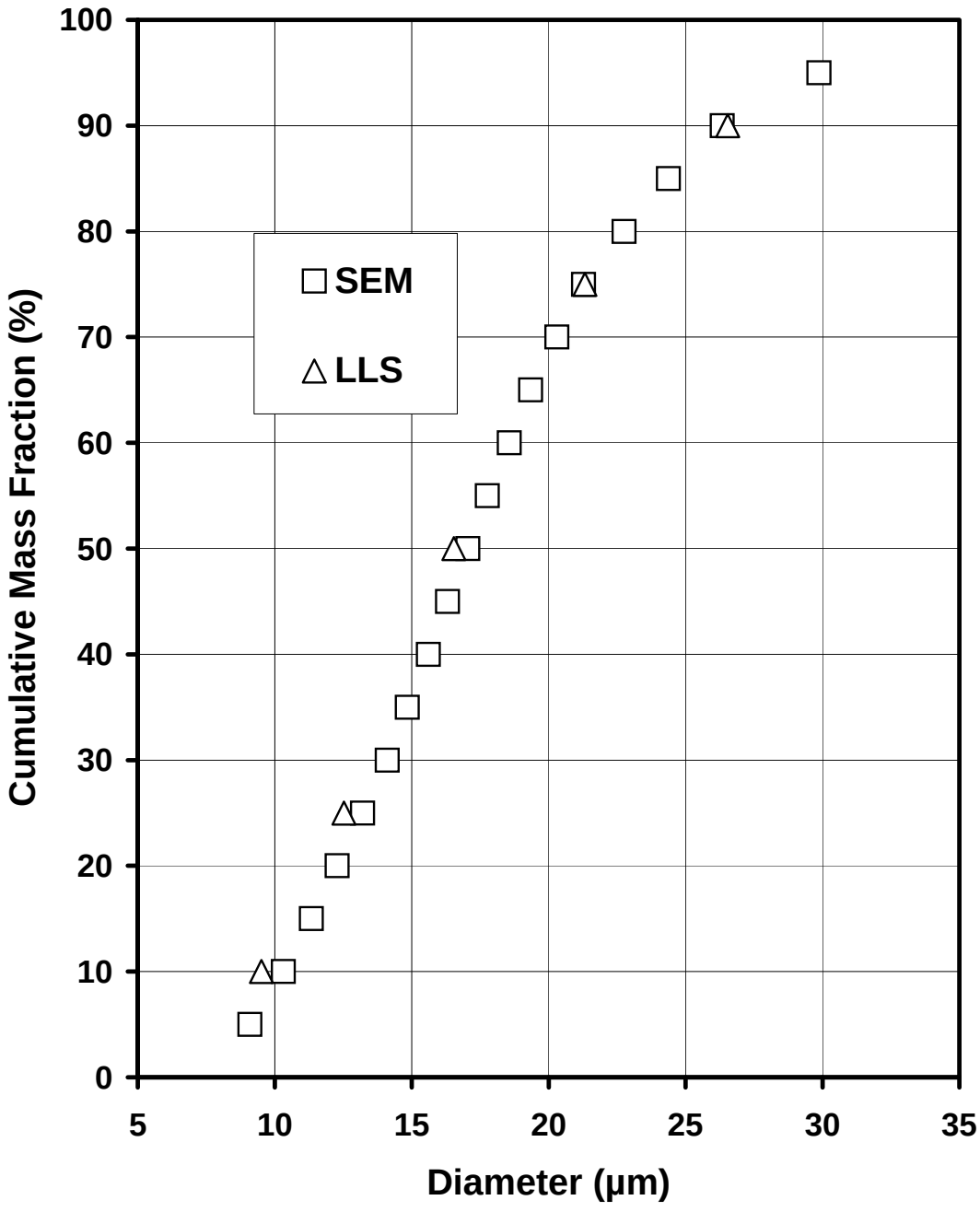

Figure 1. SRM 1984 Size Determination by SEM and LLS

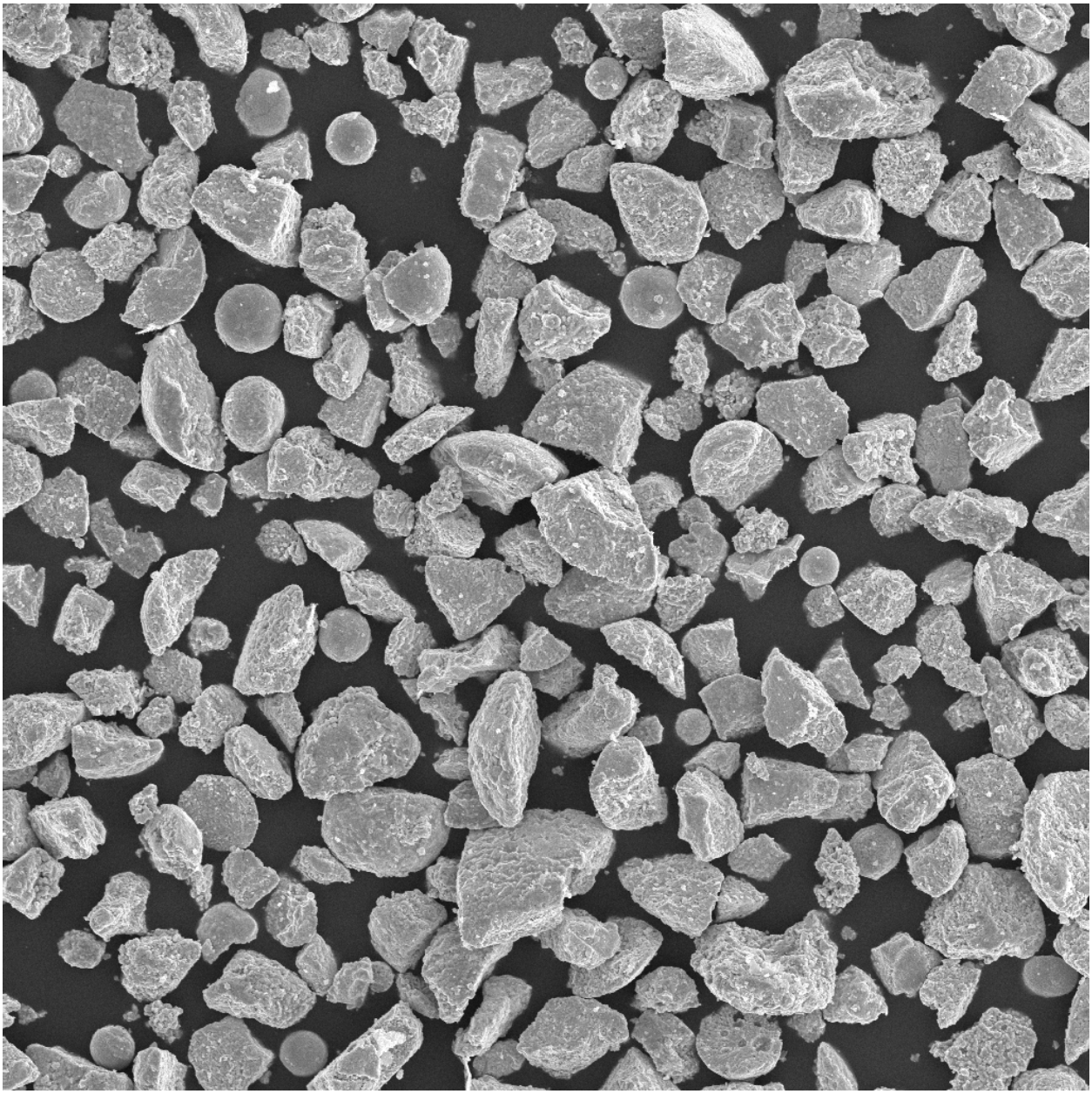

Figure 2. SEM Micrograph of SRM 1984 Powder