

MIL-Z-81572(AS)
26 January 1968

MILITARY SPECIFICATION

ZIRCONIUM OXIDE, LIME-STABILIZED,
POWDER AND ROD, FOR FLAME SPRAYING

This specification has been approved by the Naval Air
Systems Command, Department of the Navy.

1. SCOPE

1.1 Scope - This specification covers the requirements of lime-stabilized zirconium oxide (zirconia) as powder or rod for use with flame spraying processes.

1.2 Classification -

1.2.1 Types, grades and classes - Lime-stabilized zirconium oxide (zirconia) shall be furnished in the following types, grades and classes, as specified (see 6.2):

Type I - Powder (see 3.1.1)

Grade A - Sintered (see 3.1.1.1)

Class 1 - For combustion (oxy-acetylene) flame spraying

Class 2 - For plasma-arc flame spraying

Grade B - Fused (see 3.1.1.2)

Class 1 - For combustion (oxy-acetylene) flame spraying

Class 2 - For plasma-arc flame spraying

Type II - Rod (see 3.1.2)

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of the specification to the extent specified herein.

FSC 6810

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SPECIFICATIONS

Federal

RR-S-366 Sieves, Standards for Testing Purposes

STANDARDS

Military

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-1233 Procedures for Determining Particle Size, Particle Size Distribution and Packed Density of Powdered Materials

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications - The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials Publications

ASTM B 214 Methods of Test for Sieve Analysis of Granular Metal Powders

ASTM C 369 Method of Test for Modulus of Rupture of Fired, Cast or Extruded Whiteware Products

ASTM D 116 Methods of Testing Vitrified Ceramic Materials for Electrical Applications

(Application for copies of A. S. T. M. standards should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103).

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3. REQUIREMENTS

3.1 Material - Zirconium oxide (zirconia) shall be a product suitable for flame spraying and so formulated as to meet the requirements specified herein. The zirconia shall be stabilized with calcium oxide (lime) so that the resultant characteristic crystal structure will be cubic.

3.1.1 Powder (Type I) - Zirconia powder (Type I) shall be either Grade A or Grade B as specified in the contract or purchase order (see 6.1.2).

3.1.1.1 Grade A - Grade A zirconia powder shall be produced by sintering to a temperature of approximately 2600° F (1425° C) without complete melting or fusing.

3.1.1.2 Grade B - Grade B zirconia powder shall be produced by fusing to slightly above the melting point of approximately 4800° F (2650° C).

3.1.2 Rod (Type II) - Unless otherwise specified in the contract or purchase order, rods (Type II) shall be produced from fused lime-stabilized zirconia.

3.2 Chemical composition - Unless otherwise specified, zirconia shall conform to the requirements for chemical composition specified in Table I when tested in accordance with 4.5.4.

TABLE I

CHEMICAL COMPOSITION, PERCENT BY WEIGHT

COMPOSITION	Type I, Grade A (Powder, Sintered)	Type I, Grade B or Type II (Powder or Rod, Fused)
Zirconium oxide (ZrO ₂) <u>1/</u>	92.0 Min.	92.0 Min.
Calcium oxide (CaO)	4.50-5.50	3.50-5.00
Magnesium oxide (MgO)	1.40 Max.	0.50 Max.
Silicon oxide (SiO ₂)	0.80 Max.	0.80 Max.
Aluminum oxide (Al ₂ O ₃)	0.60 Max.	1.00 Max.
Titanium oxide (TiO ₂)	0.40 Max.	0.40 Max.
Ferric oxide (Fe ₂ O ₃)	0.30 Max.	0.50 Max.
Other oxides, each <u>2/</u>	0.20 Max.	0.20 Max.
Total other oxides <u>2/</u>	0.60 Max.	0.60 Max.

1/ The zirconia may include a maximum of 3 percent hafnium oxide (HfO₂) determined as zirconium oxide (ZrO₂).

2/ Need not be determined.

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3.3 Loss on ignition - The loss on ignition of Type I zirconia (powder), either Grade A or Grade B, shall not be more than 0.50 percent when determined in accordance with 4.5.5.

3.4 Particle size of powder - Unless otherwise specified, the Type I zirconia (powder), either Grade A or Grade B, shall conform to the particle size requirements of Table II when determined in accordance with 4.5.6.

TABLE II

PARTICLE SIZE REQUIREMENTS OF TYPE I ZIRCONIA

SIEVE NUMBER <u>1/</u>	MICRON OPENING	PERCENT THROUGH	
		CLASS 1	CLASS 2
140	105	--	99.0 Min.
200	74	99.0 Min.	85.0 Min.
270	53	90.0 Min.	--
325	44	70.0 Min.	25.0 Max.
--	10	8.0 Max.	--

1/ Use U. S. Standard sieves conforming to RR-S-366.

3.5 Dimensions of rods -

3.5.1 Length - Unless otherwise specified, Type II zirconia rods shall be furnished in straight lengths of 12, 18, 19-1/2 and 24 inches (see 6.2). Rods shall not vary more than 1/8 inch above or below the furnished length.

3.5.2 Diameter - The diameter of Type II zirconia rods shall be as specified (see 6.2). Unless otherwise specified, rods shall be furnished in the following diameter sizes: 1/8 (0.125), 3/16 (0.1875), 1/4 (0.25) and 5/16 (0.3125) inch. Variation of the rod diameter shall be in accordance with Table III.

TABLE III

PERMISSIBLE VARIATION IN DIAMETER OF ROD

SPECIFIED NOMINAL DIAMETER - INCH	RANGE OF DIAMETER - INCH
1/8 (0.125)	0.120 to 0.126
3/16 (0.1875)	0.182 to 0.186
1/4 (0.25)	0.242 to 0.248
5/16 (0.3125)	0.304 to 0.310

3.6 Configuration of rods -

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3.6.1 When so specified (see 6.2), 3/16 inch diameter rod shall be furnished as hollow tubes with an inside diameter of 0.025 ± 0.005 inch.

3.6.2 When so specified (see 6.2), 1/4 inch diameter rod shall be furnished with eight equally spaced radial slots. Each slot shall be 0.014 ± 0.005 inch in width and 0.047 ± 0.010 inch in depth. Straightness of slots shall not be a basis for rejection if the dimensional requirements of the slots are in compliance as herein specified.

3.6.3 When so specified (see 6.2), 5/16 inch diameter rod shall be furnished with eight equally spaced radial slots. Each slot shall be 0.0125 ± 0.0025 inch in width and 0.062 ± 0.010 inch in depth. Straightness of slots shall not be a basis for rejection if the dimensional requirements of the slots are in compliance as herein specified.

3.7 Straightness of rod - The Type II zirconia rod shall be of such straightness that the maximum curvature (depth of arc) shall not exceed the allowable tolerance as specified in Table IV.

TABLE IV

TOLERANCE FOR STRAIGHTNESS OF ROD

SPECIFIED DIAMETER OF ROD-INCH	ALLOWABLE DEVIATION FROM STRAIGHT	
	IN ANY FOOT OF LENGTH -INCH	IN TOTAL LENGTH OF ROD -INCH
1/8	0.093	0.062 x length, ft.
3/16	0.042	0.042 x length, ft.
1/4, 5/16	0.031	0.031 x length, ft.

3.8 Ends of rods - The ends of Type II zirconia rod shall be finished to a 90 degree angle from the length. An allowable deviation of 5 degrees from the square shall be permitted.

3.9 Modulus of rupture - The Type II zirconia rod shall have a modulus of rupture of not less than 3000 pounds per square inch when determined in accordance with 4.5.7.

3.10 Bulk density - The bulk density of Type II zirconia rod shall be 3.85 ± 0.20 grams per cubic centimeter when determined in accordance with 4.5.8.

3.11 Porosity - The total porosity of Type II zirconia rod shall be 30 ± 5 percent when determined in accordance with 4.5.9.

3.12 Grain size - The grain size of the material in Type II zirconia rod

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shall be determined in accordance with 4.5.10. A minimum of 20 percent of the grains composing the Type II rod shall be finer than 500 microns.

3.13 Workmanship - The zirconium oxide material for flame spraying shall be processed in a manner that will yield the highest quality product necessary to meet the requirements of this specification. The powder and rod shall be uniform in composition, quality, free from impurities and other defects which would impair its usability and adversely affect its performance. Occasional dark particles in the powder (zirconium carbide) will not be cause for rejection, provided all other requirements of the specification are maintained (see 6.4). Rods shall be uniform in size, straight, smooth and free from embedded foreign matter.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection - Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Lot - Unless otherwise specified in the contract or purchase order, a lot shall consist of the zirconium oxide material of the same type, grade, and class. Rods shall be of the same length and cross-sectional area. A lot shall consist of material produced by one manufacturer under the same continuous production run, without change in ingredients, process or conditions and offered for delivery at one time.

4.3 Sampling - Unless otherwise specified and where applicable, sampling plans and procedures in the determination of the materials submitted by a supplier shall be in accordance with the provisions set forth in MIL-STD-105.

4.3.1 Samples of Type I material (powder) -

4.3.1.1 For visual examination - A random sample shall be selected from each lot in accordance with MIL-STD-105, Inspection Level II, Acceptable Quality Level 1.0 percent defective.

4.3.1.2 For chemical requirements, loss on ignition and particle size test - A random sample shall be selected from each lot in accordance with MIL-STD-105, Inspection Level S-3, acceptance number of zero.

4.3.2 Samples of Type II material (rod) -

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4.3.2.1 For visual examination, dimensional measurements configuration, straightness and squareness of ends - A random sample shall be selected from each lot in accordance with MIL-STD-105, Inspection Level II, Acceptable Quality Level 1.0 percent defective.

4.3.2.2 For chemical requirements - Four sample rods shall be selected at random from each lot for chemical requirements. A powdered specimen shall be prepared from the selected rods by conventional methods, suitable for correct applications and usages of the techniques employed for either spectrochemical or wet chemical analysis. The samples shall be free from dirt, oil, grit and other foreign matter.

4.3.2.3 For modulus of rupture, bulk density, porosity and grain size - A random sample shall be selected from each lot in accordance with MIL-STD-105, Inspection Level S-1, acceptance number of zero.

4.4 Examination -

4.4.1 Powder and rods - Sample powder and rods selected in accordance with 4.3.1.1 or 4.3.2.1 shall be visually examined for conformance with the requirement of 3.13.

4.4.2 Packaging, packing and marking - Packaging, packing, markings, weight and closure shall conform to this specification. Each filled container of powder or rod shall be weighed to determine the net weight of contents.

4.5 Tests -

4.5.1 Dimensional measurements - The sample rods selected in accordance with 4.3.2.1 shall be measured for length to determine conformance to 3.5.1. Standard micrometer methods or go-no-go gages shall be used for measuring the rods to determine conformance to 3.5.2 for diameter and conformance to 3.6 for configuration.

4.5.2 Straightness - The sample rods selected in accordance with 4.3.2.1 shall be measured to determine conformance to 3.7. The measurement shall be taken by placing a rod on a straight edge and measuring the greatest distance and the maximum depth of arc between the straight length edge of the rod and the straight edge. At the option of the supplier any other procedure such as a warpage gage may be employed for measuring of straightness. This is essentially an adjustable slope table containing three knife edges adjusted to accommodate the various lengths of rod. A bar is attached over the knife edges which can be raised or lowered to set the allowable straightness tolerances by means of stop and feeler gages. Rods are rolled down the table to pass under the tolerance bar. Rods which do not pass freely under the bar shall be rejected.

4.5.3 Squareness of ends - The sample rods selected in accordance with 4.3.2.1 shall be measured to determine conformance to 3.8. The squareness of the

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ends shall be measured for any deviation of the end area from a straight line at right-angle to the length and touching the corner.

4.5.4 Chemical composition - The samples selected in accordance with 4.3.1.2 or 4.3.2.2 shall be tested using suitable standard wet chemical or spectrographic methods to determine conformance to 3.2 and the chemical requirements of Table I. In case of dispute, the chemical analysis by wet chemical methods shall be the basis for acceptance.

4.5.5 Loss on ignition - The samples selected in accordance with 4.3.1.2 shall be tested for loss on ignition to determine conformance to 3.3. Samples shall be dried for 2 hours in an oven at 105° to 110° C (221° to 230° F) and cooled in a desiccator to room temperature. About 10 grams of material shall be weighed immediately to the nearest thousandth of a gram upon removal from the desiccator. The specimen material shall be heated to constant weight in an electric muffle furnace at 900° to 1000° C (1652° to 1832° F). The ignition loss shall be calculated from the loss in weight.

4.5.6 Particle size of Type I material -

4.5.6.1 Class 1 - The sample of zirconia powder selected in accordance with 4.3.1.2 shall be tested for particle size distribution by sieve analysis using the procedure specified in MIL-STD-1233, Method 300, except that the shaking device shall be operated for 15 minutes, or as specified in ASTM B 214. The amounts of material retained on the sieves and bottom pan shall be weighed. Weigh out accurately 0.1 to 0.2 gram of the powdered zirconium oxide from the pan (portion that passed through the No. 325 sieve) into a beaker. Add 4 to 5 drops of a 4 percent tetra sodium pyrophosphate solution ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$), and 250 milliliters of a 1 percent sodium chloride solution (NaCl), to the material in the beaker. The beaker and contents shall then be placed on a Coulter Electrical Sensing-Zone Particle Analyzer. Counts shall be performed at 5, 10, 15, 20, 25, 30, 35, and 40 microns. The direct frequency data shall be converted by plotting the particle diameter in microns versus the weight percent above stated size (cumulative weight) in order to calculate the size distribution of the material which passed through the No. 325 sieve. Calculate and correct the final results to determine conformance to Table II. At the option of the supplier, other equipment may be used for determining the precise size measurements of the fine powder if capable of determining particle sizes between 2 to 50 microns.

4.5.6.2 Class 2 - The sample of zirconia powder selected in accordance with 4.3.1.2 shall be tested for particle size distribution by sieve analysis using the procedure specified in MIL-STD-1233, Method 300, except that the shaking device shall be operated for 15 minutes or as specified in ASTM B 214. The amounts of materials retained on the sieves and bottom pans shall be weighed to calculate percentage to determine conformance to Table II.

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4.5.7 Modulus of rupture - The samples selected in accordance with 4.3.2.3 shall be tested for modulus of rupture to determine conformance to 3.9 using the method detailed in ASTM C 369. The distance between supports shall not be less than 2 inches. When testing rods with slotted configuration, random slot orientation shall be used. In the calculation of modulus of rupture, the diameter of the specimen shall be calculated on the basis of a solid rod, neglecting corrections for center hole or rod slots if such configurations are used.

4.5.8 Bulk density - Samples of rods selected in accordance with 4.3.2.3 shall be tested for bulk density to determine conformance to 3.10. Each test specimen shall weigh from 2 to 25 grams. Test specimens shall be dried for 2 hours in an oven at 105° to 110° C (221° to 230° F) and then cooled in a desiccator to room temperature. Each specimen shall be weighed immediately to the nearest thousandth of a gram upon removal from the desiccator. The volume of the rod shall be calculated from actual rod dimensions, that is, solid shape value less any applicable hole or slot volume if such configuration is used. The bulk density of a specimen is the quotient of its dry weight in grams divided by the exterior volume in cubic centimeters.

4.5.9 Porosity - Samples selected in accordance with 4.3.2.3 shall be tested for total rod porosity to determine conformance to 3.11 in accordance with ASTM D 116. The specimen used for bulk density (see 4.5.8) may be used for total porosity.

4.5.10 Grain size - Samples selected in accordance with 4.3.2.3 shall be tested for size of the grains in the rod to determine conformance to 3.12. A thin specimen of the rod material shall be sectioned and examined under a petrographic microscope.

4.6 Rejection and retests -

4.6.1 Rejection - When one or more test specimens fails to meet any of the tests required by this specification, the lot represented by the specimen or specimens shall be subjected to rejection, except as otherwise provided by MIL-STD-105.

4.6.2 Resubmitted lots - Paragraph 6.4 of MIL-STD-105 shall apply except that a resubmitted lot shall be inspected using tightened inspection. For visual examination where the original acceptance number was zero, a sample size represented by the next higher sample size code letter shall be chosen.

5. PREPARATION FOR DELIVERY

5.1 Application - The requirements of Section 5 apply only to direct purchases by or direct shipments to the Government.

5.2 Packaging - Unless otherwise specified, the lime-stabilized zirconium oxide for flame spraying shall be packaged Level C to afford the minimum degree of

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protection necessary to prevent deterioration or damage during shipment under normal environmental conditions and commercial modes of transportation.

5.3 Packing - Unless otherwise specified, the lime-stabilized zirconium oxide for flame spraying that requires overpacking by the carrier shall be packed Level C, in exterior type shipping containers in a manner that will insure safe transportation at the lowest rate to the point of delivery, and shall meet as a minimum, the requirements of the rules and regulations applicable to the mode of transportation selected.

5.4 Marking - In addition to any special marking required by the contract or purchase order (see 6.2), packages and shipping containers shall be marked in accordance with MIL-STD-129. In addition, all containers shall be marked with the following information:

(a) Stock number or other identification number as specified in the purchase document. (The supplier shall enter the Federal Stock Number specified in the purchase document or as furnished by the procuring activity. When the Federal Stock Number is not provided or available from the procuring activity, leave space therefor and enter the Stock Number or other identification when provided by the procuring activity.)

(b) Zirconium Oxide, Lime-Stabilized for Flame Spraying Use

(c) Specification MIL-Z-

(d) Type - Powder or Rod (as applicable)

(e) Grade - Sintered or Fused (as applicable)

(f) Class of Powder (if applicable)

(g) Manufacturer's and supplier's name(s)

(h) Lot number

(i) Date of manufacture

(j) Net weight

6. NOTES

6.1 Intended use - The lime-stabilized zirconium oxide material is intended to be used with flame spraying processes to form a coating on the surfaces of a metal. The coating will act as a thermal and insulating barrier in order to prevent or delay contact between the metal and the atmosphere. The zirconia coated metal

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is employed in heat exchangers, rocket-motor nozzles, exhaust manifolds, jet engine parts, missile parts, etc.

6.1.1 Both grades of zirconium are stabilized by introducing another oxide (calcium) into the crystal lattice of the monoclinic type to change the structure to cubic.

6.1.2 For flame spraying, either grade of the powdered zirconia may be used. The difference between the two grades depends upon the processing procedures employed by the supplier, which will only affect the chemical composition (see 3.2) and the particle hardness of the zirconia material. The properties and characteristics of the resultant flame sprayed zirconia coating will be the same, regardless of grade of powder used, if surface preparation is similar, correct flame spraying procedures are used, proper coating failure measures are taken and the coating similarly finished.

6.2 Ordering data - Procurement documents should specify the following:

- (a) Title, number and date of this specification
- (b) Type of material (see 1.2.1)
- (c) Grade and class of material, if applicable (see 1.2.1)
- (d) Special chemical composition requirements, if applicable (see 3.2)
- (e) Special particle size requirements, if applicable (see 3.4)
- (f) Length and diameter of rod, if applicable (see 3.5.1 and 3.5.2)
- (g) Configuration of rod, if applicable (see 3.6)
- (h) Minimum lot size, if applicable (see 4.2)
- (i) Whether other than Level C packaging and packing is required (see 5.2 and 5.3)
- (j) Additional markings, if necessary (see 5.4)

6.3 Control - Strict control of diameter (see 3.5.2) and straightness (see 3.7) of the Type II zirconia rods for flame spraying is required to eliminate problems of rod sticking and blowback in flame spraying.

6.4 Dark particles - There are occasional dark particles, zirconium carbide, found in zirconia powder (Type I, Grades A and B) which are almost an

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immeasurable percentage of other materials. This tramp material can be picked up and may or may not appear in the finished flame sprayed coating as occasional dark specks. These dark particles or specks in no way affect the quality and properties of either the zirconia powder or of zirconia coating (see 3.13).

(Project No. 6810-N765)

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 119-R004
INSTRUCTIONS		
This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity (as indicated on reverse hereof).		
SPECIFICATION MIL-Z-81572(AS) ZIRCONIUM OXIDE, LIME-STABILIZED, POWDER AND ROD, FOR FLAME SPRAYING		
ORGANIZATION (of submitter)	CITY AND STATE	
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?		
A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE?		
<input type="checkbox"/> YES <input type="checkbox"/> NO IF "YES", IN WHAT WAY?		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity)		DATE

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DEPARTMENT OF THE NAVY
Naval Air Systems Command
Washington, D.C. 20360

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