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Designation: F 1238 – 95 (Reapproved 1999)

Standard Specification for Refractory Silicide Sputtering Targets for Microelectronic Applications [1]

This standard is issued under the fixed designation F 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers sputtering targets fabricated from metallic silicides (molybdenum silicide, tantalum silicide, titanium silicide, and tungsten silicide). These targets are referred to as refractory silicide targets, and are intended for use in microelectronic applications.

1.2 The values stated in SI units are regarded as standard.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *raw material lot*—powder mix lot from which a number of targets is fabricated.

2.1.2 *relative density*—actual target density related to theoretical density, (see 3.1.3), stated as percent.

2.1.3 *theoretical density*—calculated density for given composition as described in 5.3.

3. Ordering Information

3.1 Orders for these targets shall include the following:

3.1.1 Type and ratio (see 4.1 and 4.2),

3.1.2 Whether low alpha grade is required, (see 4.4),

3.1.3 Minimum relative density, if other than 90 %, (see 5.1),

3.1.4 Configuration, (see 6.1),

3.1.5 Whether certification is required, (see 10.1).

4. Chemical Composition

4.1 *Type*—Targets shall be classified by the following major constituents:

4.1.1 Molybdenum silicide, (Mo/Si),

4.1.2 Tantalum silicide, (Ta/Si),

4.1.3 Titanium silicide, (Ti/Si), and

4.1.4 Tungsten silicide, (W/Si).

4.2 *Ratio*—Target composition shall be stated as the atomic ratio of silicon to metal, such as Ta/Si 2.5. Ratio tolerance shall be ± 0.1. Therefore, the acceptable range for a 2.5 target would be 2.4 to 2.6.

NOTE 1—Silicon content may be calculated from the following formula:

$$\text{Silicon, \%} = \frac{\text{Ratio} \times A}{(\text{Ratio} \times A) + B} \times 100$$

where:
A = atomic weight of silicon, and
B = atomic weight of metal, (see Table 1[2, 3, 4]).

4.3 *Impurities*—Maximum impurity levels shall conform to the requirements prescribed in Table 2.

4.4 *Low Alpha Grade*—When low alpha grade targets are ordered they shall contain a maximum impurity level of uranium and thorium as agreed upon by the supplier and the purchaser. The method of analysis for these elements shall also be agreed upon.

NOTE 2—An alternative method for defining low alpha grade targets is to specify an alpha-emission rate. Specific methodology and emission rate shall be agreed upon by supplier and purchaser.

4.5 When required by purchaser, supplier will provide a 25 g sample of material that is representative of the total production process for the particular raw material lot, (see Section 8).

5. Physical Properties

5.1 Minimum relative density shall be 90 %. Other relative densities may be specified by the purchaser.

5.2 Actual target density shall be determined by Archimedes principle or other acceptable techniques.

5.3 Theoretical density shall be calculated from the following formula:

$$\text{Theoretical density} = \frac{C + (\text{Ratio} - 2) \times A}{C/D + (\text{Ratio} - 2) \times A/E}$$

[1] This specification is under the jurisdiction of ASTM Committee F-01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputter Metallization.

Current edition approved Sept. 15, 1995. Published November 1995. Originally published as F 1238 – 89. Last previous edition F 1238 – 89 (1994) $^{\epsilon 1}$.

[2] Molybdenum disilicide and WSi_2(equilibrim tetragonal phase) densities were computed from crystal lattice parameters tabulated in "Crystal Data-Determinative Tables, Third Edition," Vol 2, U. S. Department of Commerce, National Bureau of Standards and the Joint Committee on Powder Diffraction Standards, 1973, and JCPDS Data File Number 11-195. Tantalum disilicide (hexagonal) and α-$TiSi_2$(orthorhombic) data are from Einspruch, N. G. and Larrabee, G. B., *VLSI Electronics Microstructure Science*, Vol 6, Table A.1, Academic Press, NY, NY, 1983.

[3] *Binary Alloy Phase Diagrams*, Vol 2, ASM, Metals Park, OH.

[4] Einspruch, N. G., and Larrabee, G. B., *VLSI Electronics Microstructure Science*, Vol 6, Table A.1, Academic Press, NY, NY, 1983.

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TABLE 1 Weights and Densities of Constituents [A]

Constituents	Atomic or Molecular Weight	Density (g/cm [3])
Molybdenum (Mo)	95.94	...
Silicon (Si)	28.09	2.33
Tantalum (Ta)	180.95	...
Titanium (Ti)	47.88	...
Tungsten (W)	183.85	...
Silicides:		
$MoSi_2$	152.12	6.24
$TaSi_2$	237.13	9.07
$TiSi_2$	104.06	4.13 [B]
WSi_2	240.02	9.87 [C]

[A] Molecular weights of silicides were derived from elemental atomic weights. Density of silicides were calculated from x-ray data. [2]

[B] Pertains to α-$TiSi_2$, stable at temperatures less than 1200°C [3].

[C] Pertains to the tetragonal phase, which is typical for bulk material (for example. sputtering targets) made from WSi_2 powder densified in elevated temperature manufacturing processes. Sputtered thin film may stabilize in the higher density hexagonal phase. [4]

TABLE 2 Maximum Impurity Levels, ppm

Alkalis, each (K, Li, Na)	2
	5
Refractory metals, total (Mo, Ta, Ti, W), when present as impurities	150
Iron, total	100
Other metals, total (Al, B, Ca, Co, Cr, Cu, Mg, Mn, Ni)	250
Carbon	500
Oxygen	2000

where:

A = atomic weight of silicon,
C = molecular weight of metal silicide, ($MoSi_2$, etc),
D = density of metal silicide, and
E = density of silicon, (see Table 1).

6. Dimensions

6.1 Each target shall conform to an appropriate engineering drawing.

7. Workmanship, Finish, and Appearance

7.1 There shall be no radial cracks.

7.2 There shall be no other cracks or chips on the sputtering surface.

7.3 Examination for cracks and chips shall be made by the unaided eye under good lighting conditions.

8. Sampling

8.1 Analyses for ratio, (see 4.2) and for impurities, (see 4.3 and 4.4) shall be performed on a sample that is representative of the finished product.

9. Analytical Methods

9.1 Do analysis for ratio, (see 4.2) by a technique that has a precision of ±0.5 % silicon or better.

9.2 Do analysis for impurities listed in Table 1 as follows:

9.2.1 *Alkalis*—Atomic absorption (AA) with a minimum detection limit (mdl) of 0.2 ppm.

9.2.2 *Carbon*—Combustion/infrared spectrometry, mdl of 10 ppm.

9.2.3 *Oxygen*—Inert gas fusion, mdl of 10 ppm.

9.2.4 *All Others*—AA or inductively coupled plasma (ICP), mdl of 5 ppm.

9.2.5 Other analytical techniques may be used provided they can be proved equivalent to the methods specified, and have minimum detection limits of the specified methods.

9.3 Analysis for uranium and thorium or alpha-emission rate in low alpha grade targets, (see 4.4) shall have an mdl of 20 % or less of the specified level.

10. Certification

10.1 When required by the purchaser, a certificate of analysis that represents the total production process for the particular raw material lot shall be provided for each target.

10.2 Certificate of analysis shall state the raw material lot number, ratio, impurity levels, density, and dates of target manufacture and packaging.

11. Product Marking

11.1 Each target shall be marked on a non-sputtering surface with a unique raw material lot number and a unique target number.

12. Packaging

12.1 Each target shall be vacuum or inert gas packed, and enclosed in a shipping carton that ensures target integrity during shipment.

13. Keywords

13.1 density; microelectronics; molybdenum disilicide; refractory silicides; sputtering; sputtering targets; tantalum disilicide; titanium disilicide; tungsten disilicide

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