in cooperation with the WG 'Special Materials'
of the Committee of Chemists of GDMB

European Reference Materials

CERTIFICATE OF ANALYSIS

ERM®-ED101

Silicon Nitride Powder

Certified Values		
	Certified value [1]	Uncertainty [2]
Element	Mass fraction in mg/kg	
Al	469	± 12 [3]
Ca	14.1	± 0.5 [3]
Co	43.5	± 0.8
Fe	79.5	± 1.3
Mg	4.3	± 0.4 [3]
Na	7.59	± 0.27
W	41.3	± 1.3
Element	Mass fraction in %	
C	0.162	± 0.024 [3]
N	38.1	± 0.2
Mass fraction of the β-phase of silicon nitride[4] in %		
	7.43	± 0.09

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or a different method of measurement. The values are traceable to the SI (Système International d'Unités) via calibration using sufficiently pure substances of known stoichiometry.

[2] Estimated expanded uncertainty U with a coverage factor of about k=2, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

[3] For these elements inhomogeneity contributed significantly to the uncertainty.

[4] Mass fraction of the β-phase of silicon nitride of the total crystalline silicon nitride $w(\beta\text{-}Si_3N_4, Si_3N_4^*)$:
$w(\beta\text{-}Si_3N_4, Si_3N_4^*) = [m(\beta\text{-}Si_3N_4) : m(Si_3N_4^*)] \cdot 100\%$; (symbols according to DIN 1310)
$Si_3N_4^*$ is the mass fraction of the crystalline Si_3N_4
For additional data of phase composition see certification report.

Date of issue: December 2001

All following pages are an integral part of the certificate.

After 10 years the stability of the certified values has been confirmed by control analyses. Therefore the certificate remains valid until April 2039.

The minimum sample size for chemical analysis is: see instructions for use.

NOTE

European Reference Material ERM®-ED101 was originally certified as BAM-S001. It was produced and certified under the responsibility of Bundesanstalt für Materialforschung und –prüfung (BAM) in cooperation with the Committee of Chemists of the GDMB, Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

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Indicative Values[5)]

	Indicative value [6)]	Uncertainty [7)]
Element	Mass fraction in %	
O	1.91	± 0.07

[5)] Values were not certified, but given as indicative values, when the number of accepted data sets was considered to be too low, when the spread from the round robin certification was considerably larger than the state of the practice or when only 'lower as' values were reported from the round robin certification.

[6)] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or a different method of measurement. The values are traceable to the SI (Système International d'Unités) via calibration using sufficiently pure substances of known stoichiometry.

[7)] Estimated expanded uncertainty U with a coverage factor of about k=2, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

DESCRIPTION OF THE SAMPLE

The Reference Material is available as powder, and stored in units of 50 g in glass bottles.

MEANS OF THE SERIES OF MEASUREMENTS FOR THE ANALYTICAL PROCEDURE IN ONE LABORATORY (LABORATORY MEANS)

Mass fractions in mg/kg

Line No.	Al	Ca	Co	Fe	Mg	Na	W
1	451	-	-	-	-	6.38	36.0
2	452	12.2	40.2	75.9	3.84	6.78	37.1
3	461	13	41.1	76	3.95	7.17	37.4
4	463	13.4	41.3	76.3	3.95	7.25	38.4
5	463	13.4	41.6	77.2	4.01	7.34	39.0
6	464	13.8	41.8	77.7	4.04	7.36	-
7	464	13.9	42.3	77.8	4.09	7.40	40.2
8	465	13.9	43.0	78.0	4.09	7.51	40.7
9	465	14	43	78.7	4.11	7.62	40.9
10	467	14.1	43.4	79.8	4.13	7.66	41
11	467	14.2	43.7	80	4.17	7.68	41.7
12	468	14.5	43.8	80.1	4.25	7.86	41.9
13	469	14.5	43.9	81.2	4.28	8.05	42.3
14	475	14.6	44.0	81.6	4.32	8.11	42.5
15	476	14.9	44.3	81.8	4.38	8.17	42.9
16	477	15.3	44.4	82.9	4.86	8.23	43.4
17	485	15.5	44.8	83.0	5.09	8.39	44.0
18	487	-	45.3	83.0	5.17	-	44.6
19	487	-	45.4	-	5.39		44.8
20		-	45.7				45.8
21			46.3				-
22			-				
M :	469	14.1	43.5	79.5	4.34	7.59	41.3
s_M :	10	0.8	1.7	2.5	0.46	0.53	2.7

Mass fractions in %

C	N	*O*
#	-	*1.71*
-	-	*1.72*
#	37.5	*1.77*
0.151	37.6	*1.79*
0.153	#	*1.81*
#	37.8	*1.85*
0.156	37.8	*1.92*
0.162	#	*1.93*
#	38.2	*1.93*
0.165	38.2	*1.94*
#	38.2	*1.96*
0.166	38.3	*2.01*
0.166	38.4	*2.02*
#	#	*2.04*
0.167	38.5	*2.04*
#	38.6	*2.13*
0.172	#	
#	#	
#	#	
0.162	38.1	*1.91*
0.007	0.4	*0.12*

Mass fraction w(β-Si_3N_4, Si_3N_4*) in %

Line No.	*w*	Line No.	*w*
1	7.05	23	7.53
2	7.06	24	7.55
3	7.07	25	7.57
4	7.11	26	7.62
5	7.15	27	7.62
6	7.19	28	7.64
7	7.21	29	7.72
8	7.23	30	7.74
9	7.24	31	7.76
10	7.27	32	7,87
11	7.31	33	##
12	7.31	34	7.95
13	7.36	35	##
14	7.40	36	-
15	7.42	37	-
16	7.45		
17	7.45		
18	7.47		
19	-		
20	7.49		
21	7.51		
22	7.52		
		M :	7.43
		s_M	0.24

The " - " indicates that an outlying value has been detected by a statistical test and omitted.
The " # " indicates that the measurements are based on insufficient metrological traceability (calibration by matrix CRM mainly).
The " ## " indicates that the values were withdrawn by the laboratories because measurements were carried out under non-optimized conditions.
A series of measurements comprises the values measured by one laboratory (in case of phase analysis always 5; in case of element analysis at least 5, in the normal case 6 single values). The line number should not be mistaken for the laboratory code number.

M : mean of means of data sets

s_M : standard deviation of means of data sets

numbers in *italics* are indicative values

ANALYTICAL METHOD USED FOR CERTIFICATION (ELEMENT ANALYSIS)

Element	Line No.	Analytical method
Al	1, 2, 3, 4, 5, 6, 7, 8, 10, 12, 13, 16, 17, 18	Inductively coupled plasma optical emission spectrometry (ICP OES)
	9	Electrothermal atomic absorption spectrometry (ET AAS)
	11	X-ray fluorescence analysis (XRF) after fusion decomposition
	14	Flame atomic absorption spectrometry (F AAS)
	15	Neutron activation analysis (NAA)
	19	Inductively coupled plasma mass spectrometry (ICP-MS)
Ca	(1), 2, 3, 4, 5, 6, 7, 8, 9, 12, 13, 15, 16, 17, (18)	Inductively coupled plasma optical emission spectrometry (ICP OES)
	10	Photon activation analysis (PAA)
	11	Electrothermal atomic absorption spectrometry (ET AAS)
	14	Inductively coupled plasma mass spectrometry (ICP-MS)
	19	X-ray fluorescence analysis (XRF) after fusion decomposition
	(20)	Flame atomic absorption spectrometry (F AAS))
Co	(1)	X-ray fluorescence analysis (XRF) after fusion decomposition
	2, 3, 6, 7, 8, 9, 10, 12, 13, 14, 15, 18, 20, 21, (22)	Inductively coupled plasma optical emission spectrometry (ICP OES)
	4	Photon activation analysis (PAA)
	5, 19	Neutron activation analysis (NAA)
	11	Flame atomic absorption spectrometry (F AAS)
	16	Electrothermal atomic absorption spectrometry (ET AAS)
	17	Inductively coupled plasma mass spectrometry (ICP-MS)
Fe	(1), 3, 6, 7, 8, 9, 10, 11, 12, 13, 15, 16, 17, 18, (19)	Inductively coupled plasma optical emission spectrometry (ICP OES)
	2	Inductively coupled plasma mass spectrometry (ICP-MS)
	4	X-ray fluorescence analysis (XRF) after fusion decomposition
	5	Flame atomic absorption spectrometry (F AAS)
	14	Electrothermal atomic absorption spectrometry (ET AAS)
Mg	(1), 3, 4, 5, 6, 7, 10, 11, 12, 13, 14, 16, 17, 18, 19	Inductively coupled plasma optical emission spectrometry (ICP OES)
	2	Electrothermal atomic absorption spectrometry (ET AAS)
	8	Inductively coupled plasma mass spectrometry (ICP-MS)
	9	Flame atomic absorption spectrometry (F AAS)
	15	Solid sampling electrothermal atomic absorption spectrometry (SS AAS)
Na	1, 2, 3, 4, 12, 14, 15, 17	Inductively coupled plasma optical emission spectrometry (ICP OES)
	5, 13	Neutron activation analysis (NAA)
	6, 7, 9, (18)	Flame atomic absorption spectrometry (F AAS)
	8	Inductively coupled plasma mass spectrometry (ICP-MS)
	10	Electrothermal atomic absorption spectrometry (ET AAS)

	11	Photon activation analysis (PAA)
	16	Solid sampling electrothermal atomic absorption spectrometry (SS AAS)
W	1, 2, 4, 5, (6), 7, 8, 10, 11, 12, 14, 16, 17, 18, 19, 20	Inductively coupled plasma optical emission spectrometry (ICP OES)
	3, 13	Neutron activation analysis (NAA)
	9	Photon activation analysis (PAA)
	15	Inductively coupled plasma mass spectrometry (ICP-MS)
	(21)	X-ray fluorescence analysis (XRF) after fusion decomposition
C	(1)	modified combustion analysis
	2, (19)	combustion analysis /coulometry
	(3), 4, 5, (6), 7, 8, (9), 10, (11), 12, 13, (14), 15, (16), 17, (18)	combustion analysis /IR
N	(1)	modified carrier gas hot extraction
	(2), 4, (5), 6, 7, (8), (14), 16, (17), (18), (19)	carrier gas hot extraction
	3, 9, 10, 11, 12, 13, 15	volumetric determination
O	1-16	carrier gas hot extraction

Line numbers in parenthesis apply to values not used in the calculation of the certified value.

ANALYTICAL METHOD USED FOR CERTIFICATION (PHASE ANALYSIS)

Line No.	Analytical Method (Evaluation using the Rietveld method)
21, 22	Neutron diffraction with a spallation source and time-of-flight measurement
1, 7, 37	Neutron diffraction with constant wavelength
2-6, 8-20, 23-36	X-ray powder diffraction

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SAFETY INFORMATION

1. First aid measures
   Skin contact: wash affected areas with plenty of water immediately.
   Eye contact: rinse out with plenty of water for adequate time with the eyelid held wide open. Consult a physician (Get medical aid).
   Ingestion of product: induce vomiting, consult a physician.
2. Accidental release measures
   Person-related precautionary measures: Avoid dust generation and accumulation.
   Procedures for cleaning/absorption: Clean up spills mechanically; Avoid dust generation.
3. Handling
   Avoid generating dusty conditions, if necessary provide local ventilation.
4. Exposure control and personal protective equipment
   Respiratory protection: With dust generation use particle filter, e.g. DIN 3181 P 1
   Hand protection: protective gloves

Eye protection: safety goggles

5. Disposal considerations
   Unused material: Recycle, if possible. Contact producer. Or: Disposal in accordance with local legal provisions.

INSTRUCTIONS FOR USE

The minimum of the sub-sample mass for carrying out the determination of the element contents of Al, Ca, Co, Fe, Mg, Na, W is 250 mg and for C, N, (O) 500 mg. The effectively analysed minimum sample mass for the determination of the mass fraction of the β-phase is 15 mg. The bottle containing the CRM should be opened only if the humidity in the room is below 70%. The opening duration of the bottle should be as short as possible. The lid of the bottle containing a special sealing gasket should be locked tightly immediately after usage. To ensure a representative sub-sampling of the necessary sample amount the bottle containing the CRM should be carefully rotated in different directions for about two minutes before taking the sample. Shaking movements should be avoided to prevent agglomerations. Each sub-sample has to be taken separately. For subsequent elemental analysis the sample has to be treated thermally at (135 ± 5) °C for 12 hours to achieve defined starting conditions. For the determination of metallic analytes the completeness of the required pressure digestion has to be verified.

STORAGE

The material should be stored at ambient conditions in a dry and dust-free environment.

STANDARDS AND REGULATORY INFORMATION

Standards

- DIN 51089/1 "Bestimmung von Stickstoff in Si_3N_4 mittels Trägergas-Heißextraktion"
- DIN 51089/2 "Bestimmung von Stickstoff in Si_3N_4 nach Schmelzaufschluss"
- DIN 51089/3 "Bestimmung von Stickstoff in Si_3N_4 mit der Kjeldahl-Destillation"
- JIS R 1603-1994 "Methods for Chemical Analysis of Fine Silicon Nitride Powders for Fine Ceramics"

Guidelines

- "Bestimmung von Aluminium, Calcium, Eisen, Magnesium und Natrium in Siliciumnitrid", in: Handbook "Analyse der Metalle", 2nd supplement to I Schiedsanalysen, II Betriebsanalysen, GDMB-Informationsgesellschaft mbH, Clausthal-Zellerfeld, 1992, pp. 239-241.
- "Bestimmung von Kohlenstoff in Siliciumnitrid", ib. pp. 242-243.
- "Bestimmung von Stickstoff in Siliciumnitrid", ib. pp. 244-245.
- "Bestimmung von Sauerstoff in Siliciumnitrid durch Trägergas-Heißextraktion", ib. pp. 246-248.

References

R. Matschat, K. Meyer, H.-J. Heinrich, H. Scharf; "Chemical Analysis of Metallic Impurities in Alumina, Aluminium Nitride, Silicon Nitride and Silicon Carbide", in: A. Jillavenkatesa, G.Y. Onoda (Editors), Advances in Process Measurements for the Ceramic Industry, The American Ceramic Society, Westerville, 1999, pp. 107-126.

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