National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2061

TiAl(NbW) Alloy for Microanalysis

This Standard Reference Material (SRM) is intended for microanalysis of titanium (Ti) and aluminum (Al) in aerospace components and materials with a similar matrix. A unit of SRM 2061 consists of a single cube of the alloy approximately 2 mm × 2 mm × 2 mm.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST. The uncertainty listed with the value is an expanded uncertainty (k = 2, 95 % confidence interval [1]) calculated in accordance with the ISO and NIST Guides [2].

Certified values for titanium (Ti), aluminum (Al), niobium (Nb), and tungsten (W), expressed as mass fractions [2], are provided in Table 1. The results are expressed as the certified value ± an expanded uncertainty. The certified values are the unweighted averages of the concentrations determined by wavelength dispersive X-ray fluorescence spectrometry (WD-XRF) and inductively-coupled plasma optical emission spectrometry (ICP-OES) [3]. The expanded uncertainties for Ti, Al, Nb, and W include the uncertainties attributable to the heterogeneity of these elements in addition to the uncertainties from the two quantitative methods.

Heterogeneity testing on the micrometer scale (point-to-point, within specimens) was performed with wavelength dispersive electron probe microanalysis (WD-EPMA) while the bulk heterogeneity assessment (specimen to specimen) was performed with WD-EPMA and WD-XRF. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [4].

Table 1. Certified Values with Expanded Uncertainties for SRM 2061

Element	Mass Fraction (%)		
Titanium	53.92	±	0.34
Aluminum	30.31	±	0.31
Niobium	10.78	±	0.10
Tungsten	4.38	±	0.11

Expiration of Certification: The certification of this SRM is valid until **01 January 2029**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). However, the certification will be nullified if the SRM is damaged or contaminated.

Coordination of the technical measurements for certification was accomplished under the direction of R.B. Marinenko of the NIST Surface and Microanalysis Division.

Heterogeneity and microheterogeneity testing was performed by R.B. Marinenko and J. Kessler of the NIST Surface and Microanalysis Science Division and by R. Herrera-Basurto, Guest Researcher from Centro Nacional de Metrologia, Querétaro, Mexico.

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Certificate Issue Date: 16 May 2005

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WD-XRF analytical measurements and specimen to specimen heterogeneity testing for certification of this SRM were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Analytical Chemistry Division. ICP-OES analytical measurements for certification were performed by L.L. Yu and T.A. Butler, also of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the Measurement Services Division.

Information Values: An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values for oxygen, and nitrogen are provided in Table 2. The oxygen and nitrogen values are based on inert gas fusion (IGF) analysis.

Table 2. Information Values for SRM 2061

Element	Mass Fraction (%)
Oxygen	0.232
Nitrogen	0.004

Stability: This material is considered to be stable during the period of 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.

PREPARATION, HETEROGENEITY, AND ANALYSIS[1]

Material Preparation: The material for SRM 2061 was fabricated at Wright-Patterson Air Force Base (Dayton, OH). It was extruded in the form of a 2.4 cm diameter rod that was annealed at 1370 °C to produce only the α-phase of the alloy. The rod was cut into 23 disks, 2 mm thick, that were marked on the unpolished side with a number and letter to identify their original locations in the rod. These disks were polished at NIST on one side and cleaned for EPMA heterogeneity testing. Upon completion of the heterogeneity tests, six of the disks were sliced into 2 mm × 2 mm × 2 mm cubes with a diamond saw. Most of the cubes were retained for use as SRM 2061 for microanalysis while a few were used for quantitative analyses by WD-XRF, ICP-OES, and IGF. The remaining disks were retained for use as SRM 2062.

Heterogeneity: Each of the 23 disks were tested with WD-EPMA for heterogeneity using three independent sampling strategies:

1. 40-point traverses using 2 μm steps – 2 traverses normal to one another per specimen
2. 20 random point samplings per specimen
3. Nested design sampling using 3 duplicate readings on 4 randomly selected points per specimen to determine the variances for the between specimen and the between points heterogeneities [5].

The total uncertainties determined from test No. 3 are included in the certified uncertainties assigned to each element. In addition, 15 disks were tested for between specimen heterogeneity with WD-XRF.

Analysis: For WD-XRF analysis [6], acid digestion of single cubes of the alloy (6 total, approx. 40 mg each) was followed by borate fusion with a pre-fused flux (50:50 mix of $Li_2B_4O_7$ and $LiBO_2$). Calibration was accomplished using synthetic calibrants designed to match the fused unknowns and prepared from high-purity compounds. The K $L_{2,3}$ (Kα) X-ray lines for Al, Ti, and Nb and the L_3-$M_{4,5}$ (Lα) lines for W were used in the analysis.

[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.

For ICP-OES analysis [6], single cubes of the alloy were digested in HCl and HF. Manganese was used as an internal standard. For each element, calibration was accomplished with six matrix-matched standards and a linear calibration model. The analytes Al, Ti, Nb, W, and the internal standard, Mn, were measured side-on with respect to the plasma at 394.401 nm, 337.279 nm, 269.706 nm, 239.708 nm, and 260.568 nm, respectively. Analyses were done on two different days and averaged.

Oxygen and nitrogen were analyzed by Luvak, Inc. (Boylston, MA) using IGF. Two of the 2 mm × 2 mm × 2 mm cubes were used for duplicate analyses of both elements.

INSTRUCTIONS FOR USE[2]

Storage: The alloy should be stored in a closed container such as a plastic box, in a cool, dry location, preferably a desiccator, to avoid dust or moisture contamination.

Use: To relate analytical determinations to the values on this certificate of analysis, the specimen must be mounted in an appropriate medium such as epoxy and polished for analysis in the electron microprobe or scanning electron microscope. Appropriate conduction of electrons from the specimen to ground must be provided to avoid charging. Other microanalytical techniques may require different preparative procedures for use. The specimen should be handled with clean, powder-free gloves to avoid contamination with body oils and foreign matter. Avoid contact of any kind with the polished surface once it is clean and dry. If repolishing is necessary, particles of the polishing material may adhere to or become imbedded in the surface. Most can be removed with Scotch® Removable Magic® Tape or similar transparent ("invisible") tape followed by thorough rinsing with heptane to ensure removal of any remaining tape adhesive. Once polished and cleaned, the specimen may require occasional repolishing depending upon how often it is used and how much electron beam contamination occurs.

Minimum Sample Quantity: Based on the microheterogeneity assessment, the sampling of at least 10 different randomly selected 1-μm points is recommended for Al, Ti, and Nb. A 20-point sampling is recommended for W.

REFERENCES

[1] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

[2] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H-K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol.; Vol. 105, pp. 571–579 (2000).

[4] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260–136, p.16; U.S. Government Printing Office: Washington, DC; (2000).

[5] Marinenko, R.; Leigh, S.; *Heterogeneity Evaluation of Research Materials for Microanalysis Standards Certification,* Microsc. Microanal. Vol. 10, pp 491–506 (2004).

[6] Sieber, J.R; Yu, L.L.; Marlow, A.F.; Butler, T.A.; *Traceability and Uncertainty in Alloy Analysis by Borate Fusion and XFR*; X-Ray Spectrom., Vol. 34, pp. 153-159 (2004).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

[2]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.