National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2842

Semiconductor Thin Film: $Al_xGa_{1-x}As$ Epitaxial Layers

(Al mole fraction x near 0.30)

Serial Number: SAMPLE

This Standard Reference Material (SRM) is intended for use as a reference standard for analytical methods that measure the composition of thin films, such as electron microprobe analysis (EMPA), photoluminescence (PL), auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). A unit of SRM 2842 consists of an epitaxial layer of $Al_xGa_{1-x}As$ with certified Al mole fraction x grown on a gallium arsenide (GaAs) substrate mounted to a stainless steel disk by the use of adhesive tape. Each unit is sealed in a Mylar envelope containing a nitrogen atmosphere. Proper use of the SRM as a comparison standard depends on the analytical method (see "Measurement Conditions and Procedures" and NIST Special Publication 260-163 [1]).

Certified Aluminum Value: 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 [2]. The certified value for the aluminum (Al), expressed as a mole fraction, is provided in Table 1. The certified value is based on a confirmed correlation between Al mole fraction and the energy of the peak intensity in the PL spectrum of the film [3,4]. The uncertainty of the certified value is an expanded uncertainty ($k = 2$) intended to approximate a 95 % level of confidence [5]. Two additional quality checks were performed on each SRM unit. First, the molecular beam epitaxy growth system was monitored during the growth of the specimen. For acceptance as an SRM, the Al mole fraction determined from the intensity oscillations of reflection high energy electron diffraction for each unit had to agree with the certified value within its expanded uncertainty. Second, the free carrier concentration of the film had to be between 1×10^{16} cm^{-3} and 5×10^{16} cm^{-3}.

Table 1. Certified Value (mole fraction) for Aluminum in SRM 2842

Aluminum: SAMPLE ± SAMPLE

Expiration of Certificate: The certification of **SRM 2842** is valid, within the measurement uncertainty specified, until **01 August 2031**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified. Surface oxidation and contamination will increase through use and storage of the specimen. For applications that are sensitive to surface contamination, the SRM and unknown specimens will require *in situ* cleaning, typically light sputtering. The SRM should be replaced if sputtering the film causes it to visibly roughen or to show signs of selective sputtering.

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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 2842 was provided by K.A. Bertness of the NIST Applied Physics Division.

Statistical consultation was provided by C.-M. Wang of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Kristan Corwin, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 12 February 2021
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Reference Photoluminescence Peak Energy Value: A NIST reference value is a noncertified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification [2] and is provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. The reference value and expanded uncertainty ($k = 2$) for the energy of the peak intensity of the PL spectrum for this SRM composition value is provided in Table 2. The value has been corrected to an excitation volume temperature equivalent of 25.2 °C including a laser heating correction of 0.1 °C above ambient temperature.

Table 2. Reference Energy Value for the Peak Intensity of the PL Spectrum of SRM 2842

PL Spectrum: SAMPLE eV ± SAMPLE eV

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Storage and Handling: AlGaAs is a stable chemical compound but the film is subject to surface contamination and oxidation during storage and handling. The SRM should be stored in a dust-free nitrogen atmosphere or under vacuum at temperatures below 50 °C. Incidental exposure to air for transport to or use in an analysis system was not seen to produce significant contamination until such exposure exceeds thousands of hours. The SRM should be handled by the metal mounting disk with clean, nonmetallic tweezers, without contacting the semiconductor region. Particulate contamination of the semiconductor surface may be removed with deionized water or dry nitrogen flow, and users must confirm that additional contamination has not been introduced. The adhesive tape used to mount the semiconductor to the stainless steel disk is soluble in isopropanol, acetone, and other organic solvents, and use of those solvents could result in adhesive or tape particles migrating to the specimen surface. Extreme edges of the specimen surface should be excluded from analysis.

Measurement Conditions and Procedures: Analytical methods vary in their sensitivity to materials properties unrelated to the film composition. A list of potential complications is provided in Table 3. Glow discharge mass spectrometry (GDS) and secondary ion mass spectrometry (SIMS), both conventional and time-of-flight SIMS (TOF-SIMS), are included. More detailed discussion is contained in reference 1.

Table 3. Potential Confounding Factors in Comparisons Between SRM 2842 and AlGaAs Test Films of Unknown Composition.

Method	Comments on Comparison Measurements
PL	Wavelength calibration, low doping concentration, and specimen temperature correction required [3]. Thin layers may display quantum confinement energy shifts.
X-ray Rocking Curves	Not recommended for high accuracy comparisons.
AES, XPS, TOF-SIMS	Surface oxides and hydrocarbons alter results; remove surface contamination by sputtering both SRM film and test film. Monitor for selective sputtering. Sampling depth may be an issue for thin test films.
SIMS, GDS	Ignore data points from outer 10 nm of film. Monitor for selective sputtering, development of surface topography, and matrix effects.
EMPA	Compare films of similar thickness or of uniform composition over sampling depths.

REFERENCES

[1] Bertness, K.A.; Harvey T.E.; Wang, C.-M.; Paul, A.J.; Robins, L.H.; *Composition Standards for AlGaAs Epitaxial Layers;* NIST Special Publication 260-163; U.S. Government Printing Office: Washington, DC (2006); available at https://nvlpubs.nist.gov/nistpubs/Legacy/SP/nistspecialpublication260-163.pdf (accessed Feb 2021).

[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/system/files/documents/srm/SP260-136.PDF (accessed Feb 2021).

[3] Robins, L.H.; Armstrong, J.T.; Marinenko, R.B.; Paul, A.J.; Pellegrino, J.G.; Bertness, K.A.; *High-Accuracy Determination of the Dependence of the Photoluminescence Emission Energy on Alloy Composition in* $Al_xGa_{1-x}As$ *films*; J. Appl. Phys., Vol. 93, pp. 3747–3759 (2003).

[4] Bertness, K.M.; Wang, C.-M.; Salit, M.L.; Turk, G.C.; Butler, T.A.; Paul, A.J.; Robins, L.H.; *High-Accuracy Determination of Epitaxial AlGaAs Composition with Inductively Coupled Plasma Optical Emission Spectroscopy*; J. Vac. Sci. Technol., B, Vol. 24, No. 2, pp. 762–767 (2006).

[5] 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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2021); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Feb 2021).

Certificate Revision History: 12 February 2021 (Change of expiration date; editorial changes); **18 October 2011** (Extension of certification period; editorial changes); **30 January 2007** (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.