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Certificate

Standard Reference Material 2135

Ni/Cr Thin-Film Depth Profile Standard

This Standard Reference Material (SRM) is intended primarily for calibrating sputtered depth scales and erosion rates in surface analysis. It consists of nine alternating metal thin-film layers—five layers of pure chromium and four of pure nickel—on a polished silicon (100) substrate. The individual layers have thicknesses that are nominally 53 nm for the Cr and 66 nm for the Ni.

SRM 2135 is certified for total Cr and total Ni thickness by neutron activation analysis (NAA), for individual layer uniformity and Ni/Cr bilayer uniformity by Auger sputter depth profile analysis, and for individual layer thicknesses by combining the above two results. Additional noncertified information is given for relative Ni/Cr sputtering rates and yields as well as for Rutherford Backscattering Spectroscopy (RBS) measurements of single layer thicknesses.

All certified layer thickness values are expressed as mass/unit area. Because thin film densities are not equal to bulk densities, thicknesses given in units of length are not certified, but are estimates given for information purposes only.

Intended Use

Information on material composition as a function of depth at surfaces and interfaces can be obtained by monitoring the elemental surface concentration as successive surface layers are sputter-removed at known rates by ion bombardment. SRM 2135 is intended primarily to provide a means for determining sputtered depths as well as sputter erosion rates in surface analysis. Determination of sputtered depth can be made at seven depths with this one SRM using metal/metal interfaces of known spacings. The well-ordered structure of this SRM makes it useful for verifying correct instrument operation, monitoring ion beam current-density stability, and producing sputtering conditions that achieve maximum interface resolution.

Structure Description

This thin-film structure was produced by sputter depositing, in situ, first an amorphous silicon film onto the silicon substrate, followed by alternate layers of Cr and Ni with Cr forming the outermost layer, see Figure 1. Dimensions of the substrate are 1.0 x 2.54 x 0.04 cm, with the films deposited on the largest surface. The layered face is the more reflective surface.

This SRM was fabricated at the Jozef Stefan Institute, Ljubljana, Yugoslavia, under the direction of B. Navinsek.

Auger sputter depth profile analysis was performed by J. Fine and G.P. Chambers, NBS Surface Science Division.

Neutron activation analysis was performed by R.F. Fleming, NBS Inorganic Analytical Research Division.

Rutherford backscattering spectroscopy measurements were performed by D.G. Simons and M.D. Brown, Naval Surface Weapons Center, White Oak, Maryland.

Statistical consultation was provided by R.C. Paule, NBS National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed by J. Fine.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby and R.W. Seward.

Gaithersburg, MD 20899
June 26, 1985

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Certified Values

A. Total Cr and total Ni thicknesses.

The total thickness of the five Cr layers and the total thickness of the four Ni layers were determined by neutron activation analysis (NAA) using a gravimetrically calibrated reference and are given below.

Element	Total Thickness	Uncertainty
Cr	191 $\mu g/cm^2$	± 8 $\mu g/cm^2$
Ni	235 $\mu g/cm^2$	± 9 $\mu g/cm^2$

The uncertainty stated is based on the best estimate of all factors involved and corresponds to about two standard deviations of the average.

B. Individual layer uniformity and Ni+Cr bilayer uniformity (periodicity).

Layer thickness uniformity was determined by measuring the ion bombardment time required to sputter-remove a single layer. This time was obtained by monitoring the surface composition by Auger spectroscopy during sputter erosion. The uniformity of Ni+Cr bilayer removal also was determined in a similar way.

Type of Layer	Relative Layer Thickness Uncertainty*	Number of Profile Measurements (9 layers each)
Single Cr layer	± 4.8%	6
Single Ni layer	± 4.6%	6
Bilayer of Ni and Cr	± 3.0%	6

*This uncertainty is two relative standard deviations.

C. Individual layer thicknesses.

The average thickness for a single layer of a given element was obtained from the total layer thickness for that element and the number of layers present. The relative uncertainty of this average layer thickness was determined by combining the uncertainties in the total thickness and in the single layer uniformity for that element.

Element	Single Layer Thickness	Relative Layer Thickness Uncertainty*
Cr	38.2 $\mu g/cm^2$	± 2.3 $\mu g/cm^2$
Ni	58.8 $\mu g/cm^2$	± 3.5 $\mu g/cm^2$

*This uncertainty is two standard deviations.

Additional Information--Noncertified

A. Layer thicknesses in units of length.

The certified layer thicknesses could be converted into units of length (nm) if the densities of the layers were known. As these densities are not known, an approximate thickness in nm can be derived by using the known bulk densities for Cr (7.19 g cm^{-3}) and Ni (8.9 g cm^{-3}); the values so obtained are 53 nm for Cr and 66 nm for Ni.

B. Layer thickness and uniformity.

Measurements of SRM 2135 using Rutherford Backscattering Spectroscopy (RBS) give information on the single-layer thickness and uniformity of each of the Cr and Ni layers. RBS measurements of the single-layer uniformity confirm the uncertainties obtained from the sputter removal times for single layers. Layer thickness measurements obtained by RBS depend on the value of the stopping power used to calculate thickness. A 5 to 10% uncertainty in thickness can result from the uncertainty in currently available stopping power data and can limit the accuracy of this technique. Single layer thicknesses determined by RBS confirm the values certified, providing that a 5 to 10% uncertainty is assumed in the RBS measurements. The RBS result obtained for the average thickness of a single Cr layer is 8.5% smaller than its certified value, while the RBS single Ni value is 5.4% smaller than that certified.

C. Relative sputtering rates and yields of Ni and Cr using argon ion bombardment.

Ion Energy (keV)	Ni/Cr Rate	Ni/Cr Yield
1.0	1.03	1.12
2.0	1.07	1.17
3.0	1.09	1.20
4.5	1.09	1.20

Rate is expressed in units of length/time.
Yield is given as number of atoms sputtered per incident ion.

Instructions for Use

The thin-film metal layers are distinguished as the more reflective surface of this SRM. The outer Cr layer is not intended for depth calibration purposes but can be useful for ion beam current measurement and specimen alignment. Sputtered depth and rate measurement can begin at the outermost Cr/Ni interface using, for example, 50% of maximum elemental signal intensity as a reference point for layer removal measurement. Greater accuracy of sputter removal times and depths can be obtained from a periodicity measurement for the removal of a Ni+Cr bilayer pair.

An example of an Auger sputter depth profile obtained on this SRM is shown in Figure 2.

Precautions

1. The usefulness of SRM 2135 for sputter rate calibration depends on maintaining a constant ion current density at the point of analysis. Care must be taken to ensure that the ion beam current remains constant and that the beam position on the specimen remains fixed.
2. Precise determination of a reference point for layer removal measurement requires that the single layer profiles be clearly resolved (i.e., flat topped profiles) so that the sputtered interface regions are well defined.
3. Foreign surface particulates should be removed before profiling by use of clean, pressurized gas; it is not advisable to use solvents for cleaning as they can leave some residue.

The following publications provide information on the characterization and application of this SRM.

Simons, D.G., Brown, M.D., Fine, J., Andreadis, T.D., and Navinsek, B., "Rutherford Backscattering Analysis of Multilayered Cr-Ni Structures to be used for Sputtering Standards," Nucl. Instr. Meth. 218 585 (1983).

Fine, J. and Navinsek, B., "Characterization of NBS Standard Reference Material 2135 for Sputter Depth Profile Analysis," J. Vac. Sci. Technol. A3 1408 (1985).

Fine, J., Lindfors, P.A., Gorman, M.E., Gerlach, R.L., Navinsek, B., Mitchell, D.F., and Chambers, G.P. "Interface Depth Resolution of Auger Sputter Profiled Ni/Cr Interfaces: Dependence on Ion Bombardment Parameters," J. Vac. Sci. Technol. A3 1413 (1985).

Ni/Cr Multilayered Thin-Film Standard Reference Material

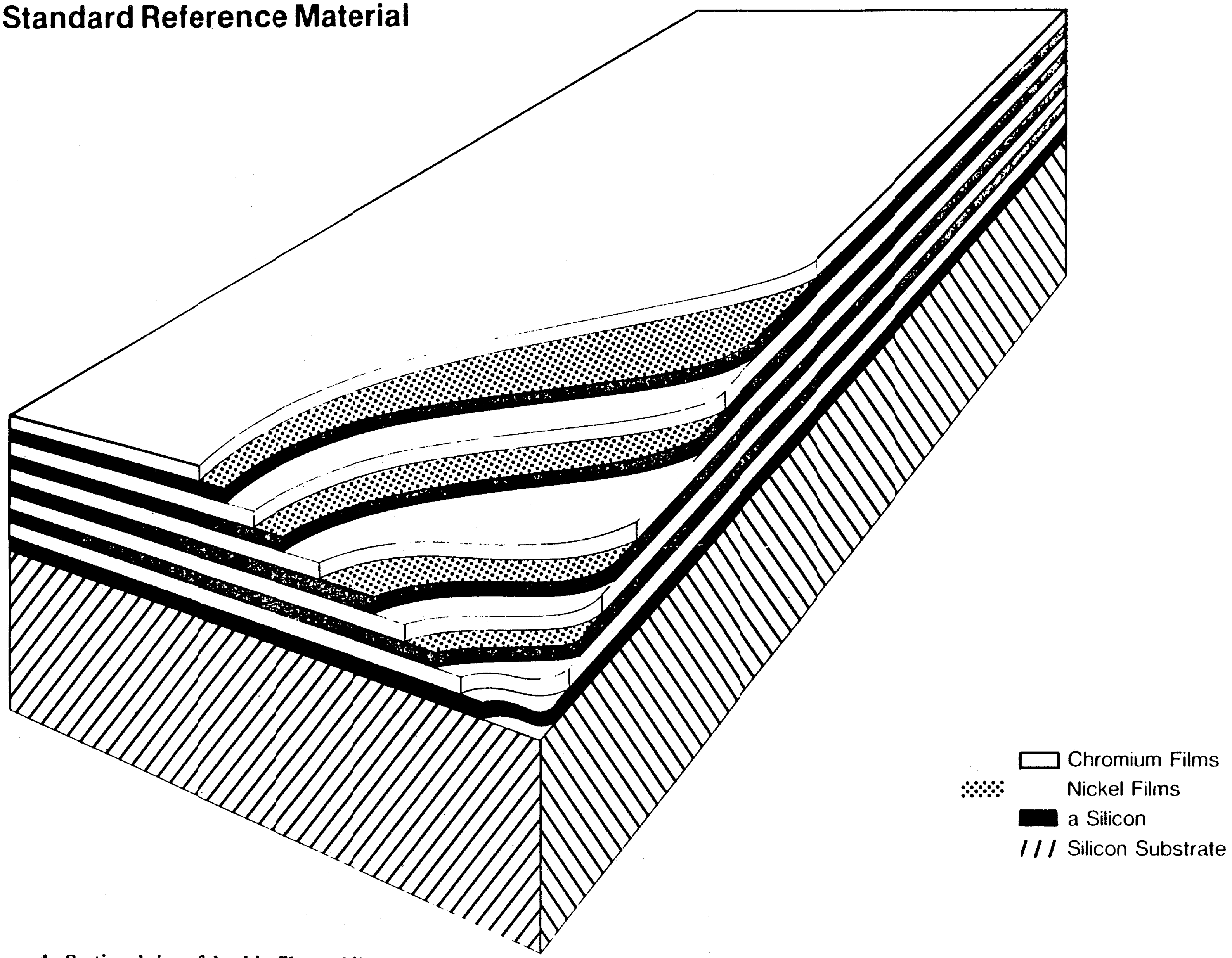

Figure 1. Sectional view of the thin-film multilayered SRM structure. The nominal thickness of each Cr layer is 53 nm and each Ni layer is 66 nm.

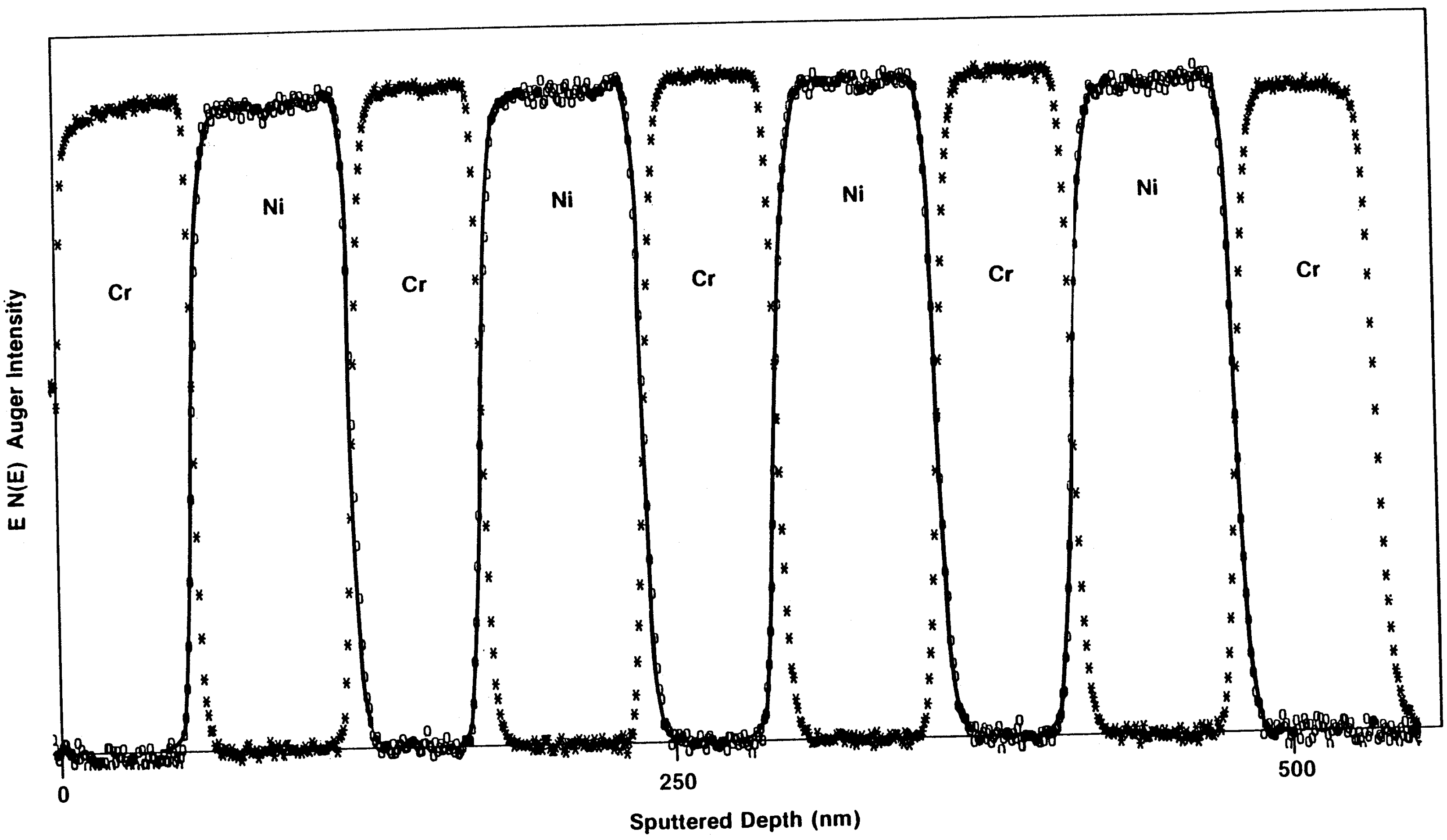

Figure 2. Auger sputter depth profile of SRM 2135 obtained using 1 keV argon ion bombardment. Total sputtering time was 16 hours; the Ni(0) and Cr(x) Auger intensities shown have been normalized.