Reference Materials 8096 and 8097
The Microelectromechanical Systems 5-in-1 Reference Materials: Homogeneous and Stable[1]

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The NIST Microelectromechanical Systems (MEMS)[2] 5-in-1 Reference Materials (RMs) were developed to assist users in validating their use of five documentary standard test methods. A Reference Material can be defined as a material whose property values are sufficiently homogeneous, stable, and well established such that the values can be used in the assessment of a measurement method. This paper presents the data that are used to characterize the RMs as homogeneous and stable.

Introduction[3]

The Microelectromechanical Systems (MEMS) 5-in-1 Reference Material (RM) (1-5) is a test chip containing test structures from which dimensional and material property measurements are extracted using five documentary standard test methods (6-10). Users can validate their use of the documentary standard test methods by comparing their measurements with NIST measurements on the same test structures.

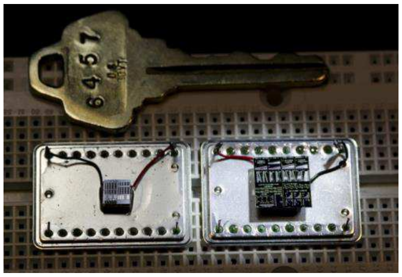

Figure 1. RMs 8096 (left) and 8097 (right)

There are two types of MEMS 5-in-1 RMs (see Fig. 1): one called the CMOS MEMS 5-in-1 Test Chip that is fabricated on a multi-user 1.5 µm complementary metal oxide semiconductor (CMOS) process followed by a bulk-micromachining etch (11) and the other called the MEMS 5-in-1 Test Chip that is fabricated using a polysilicon multi-user surface-micromachining MEMS process with a backside etch (12). These correspond to RMs 8096 and 8097, respectively. A Reference Material can be defined as a material whose property values are sufficiently homogeneous, stable, and well established such

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[2] MEMS are also referred to as microsystems technology (MST) and micromachines.

[3] Commercial equipment, instruments, or processes may be identified. This does not imply recommendation or endorsement by the National Institute of Standards and Technology (NIST), nor does it imply that the equipment, instruments, or processes are the best available for the purpose.

that the values can be used in the assessment of a measurement method (13). This paper reports homogeneity and stability results for RM 8096 and two lots of RM 8097 (Lot 95 and Lot 98).

For each RM, eight parameters and their expanded uncertainties extracted from the given RM are reported. The eight parameters are Young's modulus, residual strain, strain gradient, step height, in-plane length, residual stress, stress gradient, and thickness. The first section below entitled "Reported Expanded Uncertainty" discusses the expanded uncertainty as reported in the Report of Investigation that accompanies an RM.

Tables I to III for RMs 8096, 8097 Lot 95, and 8097 Lot 98, respectively, present the average value, P_{ave}, of each parameter as obtained from n number of RMs. These tables also include the homogeneity expanded uncertainty, U_{homog}, and the stability expanded uncertainty, $U_{stability}$, as well as the heterogeneity expanded uncertainty, U_{limit}, for each parameter as discussed in the applicable section that follows. The section entitled, "Quarterly Stability Results" presents the quarterly stability results.

Reported Expanded Uncertainty

Each NIST Reference Material (RM) is accompanied by a Report of Investigation (ROI) which includes eight NIST Reference Values (for the pre-selected test structures on the given RM) as well as the expanded uncertainty on the ROI, U_{ROI}, for each value (where k=2 gives an expanded uncertainty approximating a 95 % level of confidence (14)).

The reported expanded uncertainty on the ROI, U_{ROI}, is the expanded uncertainty obtained from the pertinent data sheet, U_{DS}, (4) and the stability expanded uncertainty, $U_{stability}$ (as calculated in the section entitled, "Stability Expanded Uncertainty") added in quadrature using the following equation:

$$U_{ROI} = (U_{DS}^2 + U_{stability}^2)^{1/2} \quad . \qquad [1]$$

Homogeneity Expanded Uncertainty

The homogeneity expanded uncertainty is used to quantify the "sameness" of the material for the given parameter. The homogeneity expanded uncertainties are calculated to be twice[4] the standard deviation, 2σ, of the parametric measurements (used to calculate the average values in Tables I to III) where each measurement is taken from a different chip. In other words,

$$U_{homog} = 2\sigma \, . \qquad [2]$$

The expanded uncertainty calculation in Eq. (1) does not include a homogeneity expanded uncertainty component, U_{homog}, because the main goal of the RM is for users to validate their use of the test methods, and the same test structure is being used by both

[4] Where k=2 gives an expanded uncertainty approximating a 95 % level of confidence.

NIST and the user. Therefore, adding a homogeneity expanded uncertainty component would make the uncertainty larger than necessary. For information purposes, the homogeneity expanded uncertainties are given in Tables I to III for RMs 8096, 8097 Lot 95, and 8097 Lot 98, respectively. Figure 2 is a plot of the residual strain values for the RM 8096 units. The average of these values, P_{ave}, is plotted on the right with the heterogeneity expanded uncertainty as discussed in the section entitled, "Limits for Heterogeneity."

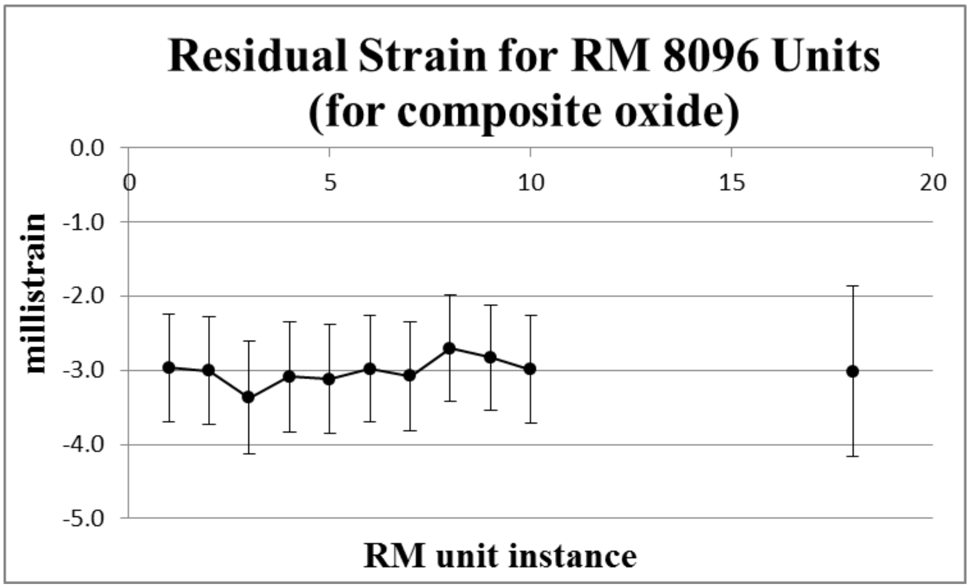

Figure 2. Residual strain values with expanded uncertainty bars, U_{ROI}, for RM 8096 units. The average value is plotted on the right with the heterogeneity expanded uncertainty (see text).

Stability Expanded Uncertainty

The stability expanded uncertainty, $U_{stability}$, is included in Eq. (1) to account for any drift in the material parameters over time. The following equation is used to calculate $U_{stability}$:

$$U_{stability} = [2(2)^{1/2} U_{DSave}] / (3)^{1/2} \quad , \qquad [3]$$

where U_{DSave} is the average of the expanded uncertainty values for n number of RMs, where each expanded uncertainty value is obtained from a data sheet for the given parameter. The values for $U_{stability}$ and n are given in Tables I to III.

Equation (3) was obtained using a two-step process: First, by determining the expanded uncertainty of the difference between two measurements, and then by assuming a uniform distribution for the stability measurements.

For the first step, to determine the expanded uncertainty of the difference between two measurements, U_D, the following equation (3) is used:

$$U_D = (U_1^2 + U_2^2)^{1/2} \quad , \qquad [4]$$

where U_1 and U_2 are the expanded uncertainties of the first and second measurements, respectively. If it is assumed that

$$U_1 = U_2 = U_{DSave} \quad , \qquad [5]$$

where U_{DSave} is the average of the expanded uncertainty values for n number of RMs, where each expanded uncertainty value is obtained from a data sheet for the given parameter, then Eq. (4) can be written as follows:

$$U_D = (2)^{1/2} U_{DSave} \quad . \qquad [6]$$

For the second step, assuming a uniform distribution for the stability measurements, the expanded uncertainty equation would be as follows:

$$U_{stability} = |P_{hi} - P_{lo}| \,/\, (3)^{1/2} \quad , \qquad [7]$$

where P_{hi} and P_{lo} are the two extreme parametric values.

If $2U_D$ is equated with $|P_{\text{hi}} - P_{\text{lo}}|$ (which assumes the initial value is an extreme value and not the midpoint such that the parametric value will either drift up or down over time (3)), then Eq. (7) can be written as follows:

$$U_{stability} = 2U_D \,/(3)^{1/2} = 2(2)^{1/2} U_{DSave} \,/\, (3)^{1/2} \quad , \qquad [8]$$

which is Eq. (3).

Equation (3) is used for the material parameters whereas $U_{stability}$ is assumed to be zero for the dimensional parameters. The stability expanded uncertainties are given in Tables I to III.

Limits for Heterogeneity

The heterogeneity limits, $P_{ave} \pm U_{limit}$, used to demonstrate fitness for purpose are the average value, P_{ave}, plus or minus U_{limit}, where U_{limit} is calculated using the following equation:

$$U_{limit} = x \, U_{DSave} \quad , \qquad [9]$$

where x is a suggested multiplier. The values for U_{limit}, x, and n are given in Tables I to III. As mentioned in the section entitled, “Homogeneity Expanded Uncertainty,” Figure 2 is a plot of the residual strain values for the RM 8096 units. The average of these values, P_{ave}, is plotted on the right with the heterogeneity expanded uncertainty. As can be seen in this figure, the residual strain values fall comfortably between these bounds.

Quarterly Stability Results

Every quarter residual strain measurements are taken on RM monitor chips (3) that are being kept under different storage conditions in order to assess the stability of the RMs. The RM monitors are stored in N_2, a clean plastic wafer carrier, or a wooden box with black foam padding. Figures 3 to 5 give the stability measurements to date for RMs 8096, 8097 Lot 95, and 8097 Lot 98, respectively.

Figure 3 of RM 8096 for the composite oxide layer (3) is particularly interesting. For optimal parametric stability, the RMs are stored in a dust-free inert atmosphere or under an oil-free vacuum. The results for Chip 1 (stored in N_2) show that the residual strain is fairly constant as a function of time. For Chip 2 (stored in a wooden box since the 4th quarter) and Chip 3 (stored in plastic since the 4th quarter) the absolute value of the residual strain appears to be increasing with time. The storage for Chip 3 was changed from dirty plastic storage to clean plastic storage after being in dirty plastic for one quarter and seeing a large increase in the absolute value of the residual strain. It is believed that contaminants (such as plasticizers) cause this increase in the absolute value of the residual strain. If the absolute value of the residual strain continues to increase with time for Chips 2 and 3, the quarterly stability results for these chips are expected to fail in two-and-a-half to three years from when they were placed in their current storage location. (The expiration date of the Reports of Investigation takes this into consideration.) They fail when the following equation is satisfied:

$$D > U_D \ , \qquad [10]$$

where D is the positive difference between the measurement under consideration and what can be considered the initial measurement and U_D is calculated using Eq. (4), where U_1 is the expanded uncertainty of what can be considered the initial measurement and U_2 is the expanded uncertainty of the measurement under consideration.

Figures 4 and 5 for the second polysilicon layer (P2) currently do not reveal any trends for the different storage conditions.

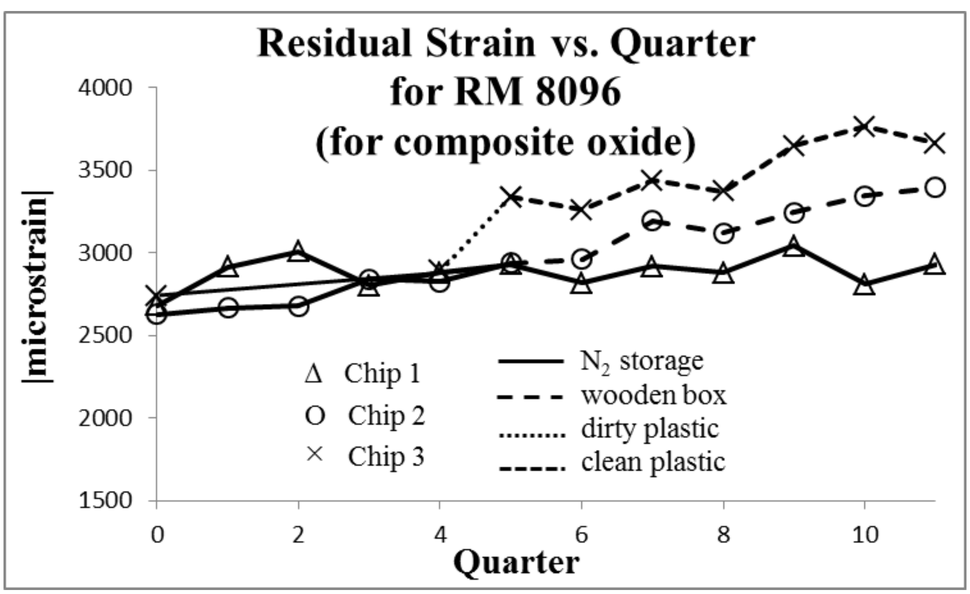

Figure 3. Stability results for RM 8096

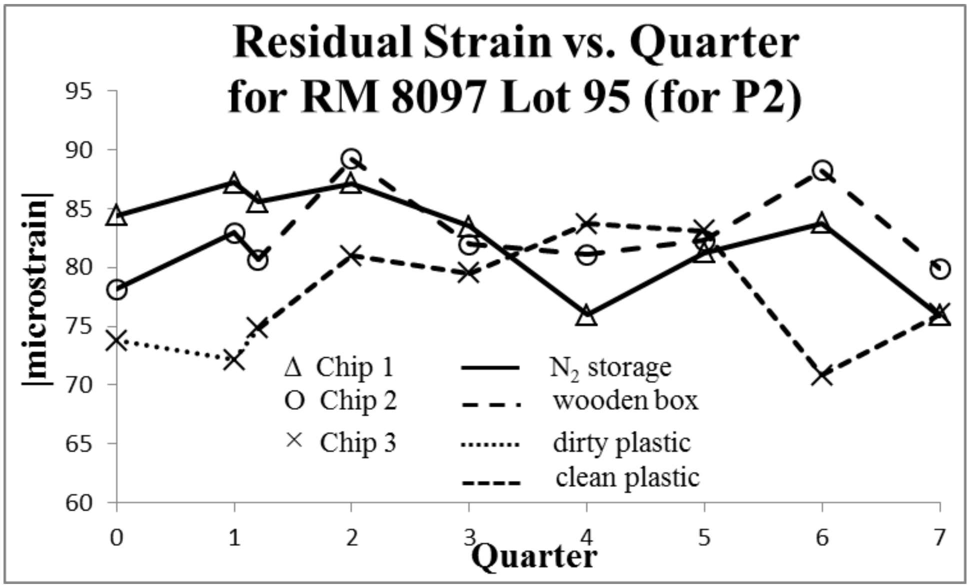

Figure 4. Stability results for RM 8097 Lot 95

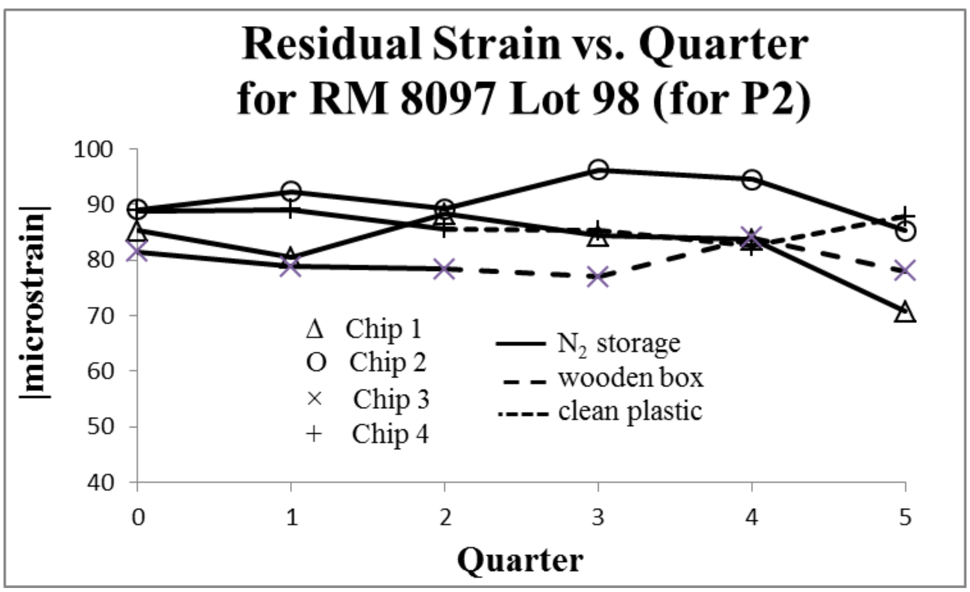

Figure 5. Stability results for RM 8097 Lot 98

Table I. For RM 8096, Average NIST Reference Values with Expanded Uncertainties and Limits for a Composite Oxide Layer, Except Where Noted

Measurement	P_{ave} (n=10)	U_{homog}	$U_{stability}$	$P_{ave} \pm U_{limit}$	
				x	U_{limit}
1. Effective Young's modulus (GPa), E	59.9	5.1	15.4	2	18.9
2. Effective residual strain ($\times10^{-3}$), ε_r	–3.02	0.35	0.62	3	1.15
3. Effective strain gradient (mm^{-1}), s_g	0.840	0.256	0.134	5	0.410
4. Step height (µm),[a] $step1_{AB}$	0.498	0.105	0.0	1.5	0.185
5. In-plane length (µm),[b] L (at 25×)	202.0	0.9	0.0	1.3	4.0
6. Effective residual stress (MPa), σ_r	–181	22	60	2	73
7. Effective stress gradient (TPa/m), σ_g	50.3	15.0	15.2	3	28.0
8. Thickness (µm), t_{oxide}	2.68	0.14	0.0	2.5	0.50

[a]This is a metal2-over-poly1 step from active area to field oxide.
[b]This measurement is taken between two metal2 lines.

Table II. For RM 8097 Lot 95, Average NIST Reference Values with Expanded Uncertainties and Limits for the Second Polysilicon Layer, Except Where Noted

Measurement	P_{ave} (n=12)	U_{homog}	$U_{stability}$	$P_{ave} \pm U_{limit}$	
				x	U_{limit}
1. Effective Young's modulus (GPa), E	127	8	33	2	40
2. Effective residual strain ($\times10^{-6}$), ε_r	–83.3	18.6	12.6	5	38.7
3. Strain gradient (m^{-1}), s_g	17.7	5.5	9.4	3	17.2
4. Step height (µm),[a] $step1_{AB}$	–0.608	0.121	0.0	1.3	0.204
5. In-plane length (µm), L (at 5×)	999.9	1.5	0.0	1.3	4.9
6. Effective residual stress (MPa), σ_r	–10.6	2.7	3.2	2.5	4.8
7. Effective stress gradient (TPa/m), σ_g	2.24	0.69	1.32	2.5	2.03
8. Thickness (µm), α	1.38	0.10	0.0	1.2	0.42

[a]This is a step from poly1 to poly2 if it is negative.

Table III. For RM 8097 Lot 98, Average NIST Reference Values with Expanded Uncertainties and Limits for the Second Polysilicon Layer, Except Where Noted

Measurement	P_{ave} (n=27)	U_{homog}	$U_{stability}$	$P_{ave} \pm U_{limit}$	
				x	U_{limit}
1. Effective Young's modulus (GPa), E	139	4	37	2	45
2. Effective residual strain ($\times10^{-6}$), ε_r	–83.1	16.3	12.2	5	37.4
3. Strain gradient (m^{-1}), s_g	14.2	9.0	7.7	3	14.1
4. Step height (µm),[a] $step1_{AB}$	–0.64	0.20	0.0	1.3	0.33
5. In-plane length (µm), L (at 5×)	999.7	1.8	0.0	1.3	4.8
6. Effective residual stress (MPa), σ_r	–11.6	2.3	3.5	2.5	5.3
7. Effective stress gradient (TPa/m), σ_g	1.98	1.25	1.19	2.5	1.82
8. Thickness (µm), α	1.28	0.10	0.0	1.2	0.54

[a]This is a step from poly1 to poly2 if it is negative.

Summary

Homogeneity and stability data were presented for RMs 8096, 8097 Lot 95, and 8097 Lot 98. The results show that the MEMS 5-in-1 RMs are homogeneous and stable within their stated uncertainty bounds, which is a salient feature of RMs. This assumes that the RMs are stored properly. In particular, RM 8096 should be stored in an inert atmosphere to help prevent contamination of the composite oxide beams, which could cause an increase in the absolute value of the residual strain. Improper storage conditions can nullify the Report of Investigation that accompanies an RM.

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2. J. Cassard, J. Geist, M. Gaitan, and D. G. Seiler, *Proceedings of the 2012 International Conference on Microelectronic Test Structures*, ICMTS 2012, San Diego, CA, pp. 211-216, March 21, 2012.
3. J. M. Cassard, J. Geist, T. V. Vorburger, D. T. Read, M. Gaitan, and D. G. Seiler, NIST SP 260-177, February 2013.
4. The data analysis sheets, SP 260-177, and details associated with the MEMS 5-in-1 can be found at the MEMS Calculator website (Standard Reference Database 166) accessible via the NIST Data Gateway (http://srdata.nist.gov/gateway/) with the keyword “MEMS Calculator.”
5. Contact the NIST Office of Reference Materials to obtain a MEMS 5-in-1 (which comes with its Report of Investigation, the pertinent data analysis sheets, the five standard test methods, and the SP 260-177) by visiting http://www.nist.gov/srm.
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11. The RM 8096 chips were fabricated through MOSIS (http://www.mosis.com) on the 1.5 µm On Semiconductor (formerly AMIS) CMOS process.
12. The RM 8097 chips were fabricated at MEMSCAP (http://www.memscap.com) using MUMPs-Plus! (PolyMUMPs with a backside etch).

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