Reference Material (RM) 8820: A Versatile New NIST Standard for Nanometrology

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ABSTRACT

A new multipurpose instrument calibration standard has been released by NIST. This standard was developed to be used primarily for X and Y scale (or magnification) calibrations of scanning electron microscopes from less than 10 times magnification to more than 300 000 times magnification, i.e., from about 10 mm to smaller than 300 nm range instrument field of view (FOV). This standard is identified as RM 8820. This is a very versatile standard, and it can also be used for calibration and testing of other type of microscopes, such as optical and scanning probe microscopes. Beyond scale calibration, RM 8820 can be used for a number of other applications, some of which will be described in this publication.

Keywords: standards, scanning electron microscope, traceability, magnification, field of view, scale, calibration

1. INTRODUCTION

Reference Material (RM) 8820 was developed primarily to be used for X and Y scale (or magnification) calibrations of scanning electron microscopes (SEMs) from less than 10 times magnification to more than 300 000 times magnification, i.e., from about 10 mm to smaller than 300 nm range instrument field of view (FOV). RM 8820 can also be used for calibration and testing of other type of microscopes, like optical and scanning probe microscopes. Beyond scale calibration, it can be used for a number of other applications such as instrument linearity measurements, optical overlay measurements, scatterometry, contamination testing and others, some of which will be described in this publication.

The overall RM 8820 was designed in collaboration with the SEMATECH Advanced Metrology Advisory Group (AMAG) and was fabricated at SEMATECH, Austin, TX. This sample is the culmination of many years of effort to provide the industry with calibration standards for laboratory and manufacturing metrology instrumentation.

1.1 History

The first lithographically produced NIST Reference Material (RM) for SEM magnification calibration was introduced to the microscopy community in 1988 [3] and released as a NIST Reference Material in 1995 as RM 8090 [4]. The RM 8090 samples were fabricated using direct write electron beam lithography and metal lift off on a silicon wafer substrate. The pattern consisted of a layer of 10 nm titanium and 40 nm palladium on a 10 x 10 mm silicon chip. The edge roughness of the 200 nm pitch structures was measured to be 5 nm or less. The lift off technique resulted in an edge roughness of approximately 4 nm.

The initial RM 8090 sample was a product of several years of research effort. The three major design constraints were 1) adequate fidelity for the smallest line structures (200 nm pitch), 2) the ability to view the sample at both high and low accelerating voltages [5], and 3) materials that are fully compatible with current CMOS production. Therefore, gold was a material that could not be used. These fundamental requirements were achieved on proof-of-concept samples which were initially fabricated by direct write e-beam lithography and manually processed at the Cornell National Nanofabrication Facility (NNF) in collaboration with Dr. Richard Tiberio [3, 6]. Following the initial proof of concept, a second batch of evaluation samples (20) was fabricated by Dr. Tiberio and NIST circulated them to the community for technical comments

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and review.

The generation of a useful reference material for the United States requires the ability for NIST to stock and make available a number of samples sufficient to meet the anticipated demands of the customer base. With literally hundreds of scanning electron microscopes in use at that time, faster and more efficient ways of fabrication needed to be sought. Through the efforts of Ms Marilyn Hoy Bennett and Dr. Brian Newell, Texas Instruments (TI), Dallas, were able to convince TI to assist NIST in fabricating the first ever production batch of the reference materials. Using direct write electron beam technology and full wafer lithographic processing capabilities, approximately 150 samples were initially fabricated [7, 8, 9]. The major portion of these samples was placed into stock within the Office of Standard Reference Materials (OSRM) and a number of the samples were distributed to about 60 laboratories across the United States in an interlaboratory study [10].

1.2 Interlaboratory Study. Several of the initial RM 8090 samples were distributed to laboratories in industry, academia and government as an Interlaboratory study with the goal of assessing the impact of the RM on the customer base. Approximately 60 laboratories across the United States agreed to participate in the study (Postek, et al., 1993). The major results clearly outlined the current calibration deficiencies and need for accurate instrument calibration. Use of the new standard clearly demonstrated that scanning electron microscopes were generally mis-calibrated as much as 60 %. Even instruments within the same laboratory were as much as 15 % mis-calibrated to each other. This study opened the eyes of the SEM researchers regarding the need to calibrate properly the instrumentation both in the laboratory and manufacturing.

The first production run of RM 8090 sold out rapidly and it became necessary to look again into the technology pool for a solution. TI agreed to provide another batch of 50 samples as an interim solution. Therefore, a second release of RM 8090 samples occurred. But, during the interim between RM fabrication batches, several changes at Texas Instruments occurred resulting in the inability for TI to fabricate any additional batches of the materials. Subsequently, other companies having direct-write e-beam capabilities volunteered to assist NIST but were unable to meet the fidelity requirements in the materials needed for the sample. Other avenues for fabrication needed to be developed.

During the technology search, x-ray lithography was considered to be a viable alternative technology. X-ray lithography was potentially a very high-fidelity and rapid throughput technology lending itself to mass fabrication of the RMs. Utilizing x-ray lithography in the fabrication of the RMs would be extremely cost-effective and many samples could be made available at low cost. The complication and eventual weakness in this technique was the fabrication of the x-ray mask. The fabrication of the x-ray lithographic mask was unsuccessful and it became necessary to look elsewhere for a solution.

Several other generous attempts were made to fabricate the sample through electron beam lithography by a number of other laboratories. These attempts proved to be unsuccessful and not cost-effective. Fortunately, optical lithography continued along the path of Moore's law as a viable technology. Through the strong collaboration between the NIST Manufactiring Engineering Laboratory and SEMATECH, state-of-the art phase-shifting masks were fabricated and experiments were done to optimize the lithography needed for the fidelity of the fine lines of the RM. This was accomplished in 2008 and 100 samples were delivered to the NIST Office of Standard Reference Materials (OSRM). Since the overall pattern on the chip was markedly different than the previous RM 8090 samples, the new RM was named RM 8820.

RM 8820 was a product of the strong SEMATECH AMAG/NIST collaboration. The overall chip was designed with the intent of not only solving the NIST need for a new reference material but also the semiconductor industry's need for additional calibration and test patterns. Therefore, the four NIST patterns found on the chip are surrounded by numerous other patterns specifically designed for applications beyond just SEM calibration.

1.3 Reference Material vs. Standard Reference Material. In order to get the new standard to the industrial users as rapidly as possible, the first introduction of this standard was as an RM (an SRM version is currently being developed and will be discussed later). A Standard Reference Material (SRM) is at the pinnacle of the reference material chain. An SRM has been highly researched and all sources of uncertainty have been explored leading to as low a measurement uncertainty as possible. A Reference Material (RM) has been highly researched, but all of the sources of measurement uncertainty have not been fully explored or accounted for. Therefore, the measurement uncertainty is higher.

2.0 Materials and Methods

2.1 Fabrication. The RM 8820 samples were fabricated on 200 mm silicon (Si) wafers using 193 nm ultraviolet light lithography and a dry etch process that formed all the patterns from an amorphous Si layer deposited on the silicon substrate with a thin silicon oxide layer between the layers. This 2 nm thin SiO_2 was used as an etch stop. Before the patterns were made, measurements were carried out to determine the thickness of the amorphous Si layer. The average thickness of the amorphous Si layer was found to be 97.3 nm with a standard deviation (σ) of 1.6 nm.

All amorphous silicon patterns exhibit a natural edge unevenness or edge roughness. This is a consequence of the lithography and etch processes. For RM 8820, the processing resulted in an average edge roughness of 6 nm. This edge roughness, however, does not have a very large effect on the pitch determination if large enough segment of the lines are considered in each pitch measurement. This can be achieved by measuring the center two-thirds of the images taken at suitable magnifications. To assist the user, a pitch calculation software program is available from NIST that will calculate the pitch on hundreds or thousands of locations and give a statistical measure of the pitch values (see the description of this program later in this document).

RM 8820 was produced through collaboration with SEMATECH in Austin, TX. An Applied Materials [1] critical dimension (CD) measurement SEM using a thermally assisted field emission source was used at SEMATECH for automatic measurements of the samples prior to dicing and packaging. Measurements of multiple scans were averaged for the NIST Metrology Instrument result. The NIST Inspection Instrument used an average of 64 lines.

2.2 Packaging. Each sample is provided in a box lined with semi-sticky plastic to protect it from damage. It is important to use (preferably conductive plastic) tools to carefully remove the chip from its box. The semi-sticky plastic will let the user move the chip slowly away from the surface by gently pushing a tool under the bottom of the chip. It takes a few seconds for the semi-sticky plastic to recede and the chip then can be lifted out of the box.

2.3 Mounting. The chip is intended to be mounted by the users on the proper stub, wafer or sample holder suitable for their particular instrument. Utmost care should be taken in the handling and mounting of the sample. Spring-loaded fasteners or a very small amount of carbon conductive paste applied at two corners of the chip were found to work well.

Electron beam-induced contamination can be deposited on the sample depending on the handling, instrument cleanliness, electron beam current and accelerating voltage used. Only the user has control over these parameters. It is possible to achieve cleanliness so that after 10 minutes of continuous imaging no visible change in the quality of the image is observed or the measured value of the pitch. This may require the use of methods that are designed to clean the sample and the sample chamber and its vacuum system [11, 12].

2.4 Sample Cleanliness. Especially at high magnifications, sample cleanliness and elimination of electron or ion beam-induced contamination are essential. Unless the sample and the microscope are clean enough, (i.e., allowing for ten minutes of continuous imaging or measurement without noticeable sign of contamination), it is likely that erroneous and misleading results will be obtained. The RM artifacts were cleaned after production, but it makes sense to check and if necessary clean the sample before it gets under the beam. Later in this document there is a detailed description of a cleaning procedure that was found to be very useful to clean (even electron beam) contaminated RM 8820 samples and the vacuum space of the microscope.

3.0 Description of NIST RM 8820

The overall RM 8820 chip is composed of an aggregate of a number of patterns developed through the SEMATECH Advanced Metrology Advisory Group (AMAG)/NIST collaboration as shown in Figure 1. The overall chip was designed with the intent of solving the NIST and industrial need for a new reference material for semiconductor manufacturing. In addition, additional calibration and test patterns identified by the AMAG are included and are designed for applications beyond just SEM calibration. Therefore, the four NIST RM 8820 patterns found on the chip are surrounded by numerous other patterns. These include atomic force microscope calibra-

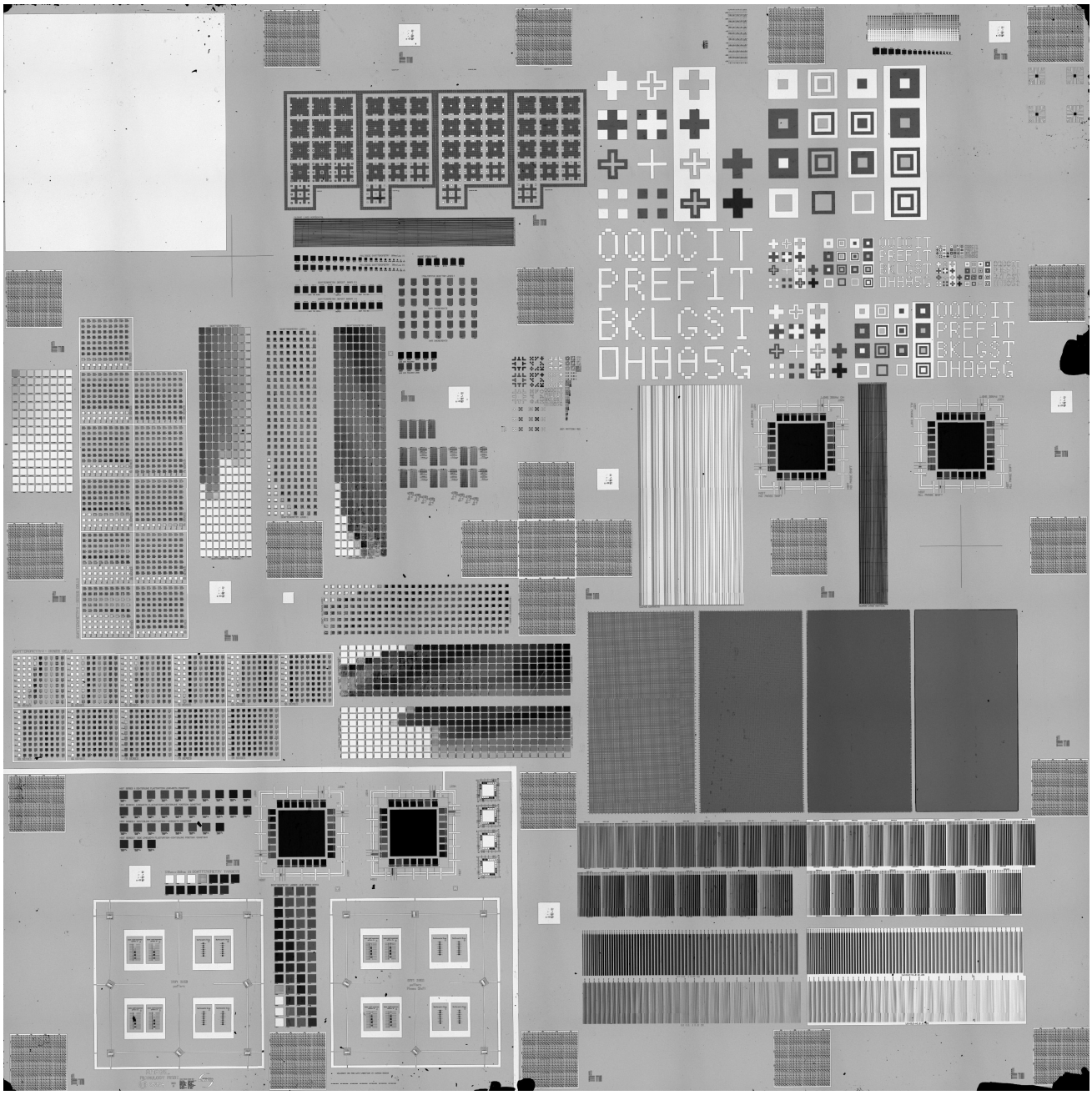

Figure 1. Overall bright field optical micrograph of the RM 8820 sample. (Courtesy of Zeiss, Inc.)

tion, optical microscope calibration, overlay metrology, line edge roughness measurement, global alignment test patterns, scatterometry test patterns, and a number of additional patterns with the general locations shown in Figure 2.

Please note: *Space constraints for this particular publication permit only room for the description of the RM 8820 pattern. A much more involved description of the entire group of patterns is being developed. It will soon be available as a NIST publication (due to its size), and copies will be available through the authors.*

3.1 Global coordinate system. The RM 8820 patterns are found within an overall square pattern on the silicon chip. Most modern SEMs and other instruments have a precise vernier system for the sample drive mechanisms whether they are manual

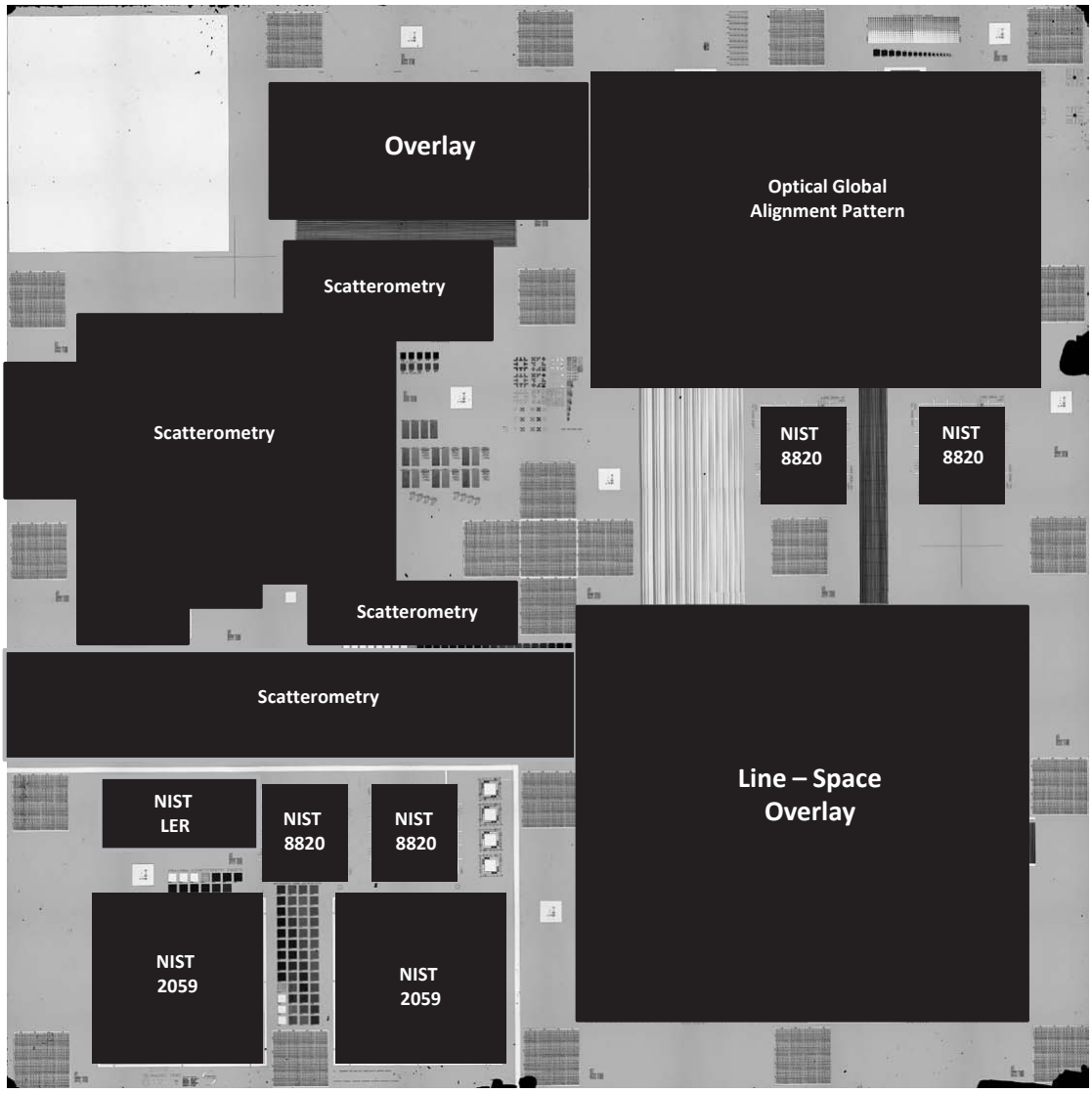

Figure 2. Identification and highlighting of some of the types of calibration and testing patterns available on RM 8820, overlaid on the bright field optical micrograph of Figure 1.

or motorized. Many of the critical dimension (CD) SEMs have automated stages and pattern recognition software. Therefore, a grid coordinate system for precise location of the patterns can be readily developed. Figure 3 shows one approach to the development of a coordinate system for locating the patterns. Here, the upper left corner is coordinate (0000, 1000); the lower left corner is coordinate (0000, 0000); the upper right corner is coordinate (1000, 1000) and the lower right corner is coordinate (1000, 0000). The 40 mm^2 chip (20 mm x 20 mm) can then be broken into 1 000 000 points; a calibration structure of interest can be precisely identified according to that grid. This system has been used to describe the calibration structures in this paper.

3.2 NIST RM 8820 SEM Calibration Pattern. The NIST SEM calibration patterns of RM 8820 have been fabricated on the 20 mm x 20 mm silicon chip as a portion of a very large set of structures as shown in Figures 1, 2 and 3. Note the locations of the four NIST scanning electron microscope calibration patterns. These patterns are 1.5 mm by 1.5 mm and are marked with letters "NIST." These are readily visible with the naked eye as small bright squares within the large chip. Like its predecessor, RM 8820 patterns have pitches ranging from 200 nm to 1.5 mm in both X and Y directions (Figure 4). Using the global coordinate system described above, the four NIST RM 8820 patterns are at coordinates 1) 733, 592;

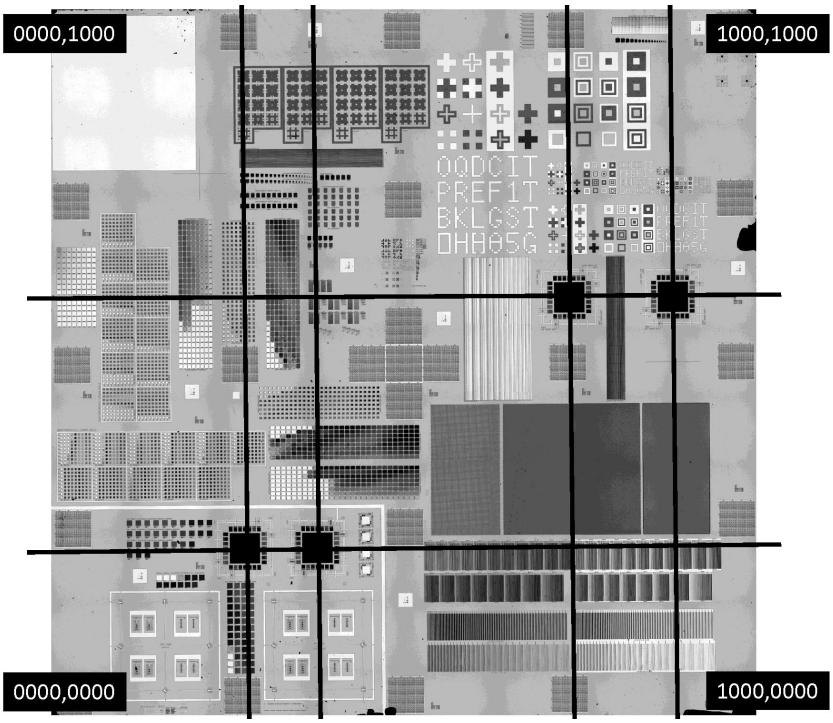

Figure 3. Optical micrograph of the overall RM 8820 with the global grid applied and the four NIST RM 8820 patterns shown at the coordinate locations described in the text.

2) 879, 592; 3) 273, 242; and 4) 376, 242 as shown in Figure 3.

The four NIST patterns are composed of two versions of the RM 8820 pattern. In one version, the structures are separate from each other; in the other version, all patterns are electrically connected to each other. This makes it possible to connect these patterns to ground potential, if that is deemed by the user to be useful. The measurements done for quality assessment were done on the non-connected patterns. There are two pairs of the connected and non-connected patterns, which all can be used for scale calibration purposes. Within each RM 8820 pattern, there are 4 sets of calibration patterns (2 in the X direction and 2 in the Y direction). In the center of all of the RM 8820 patterns, there is a large area of structures for focusing and astigmatism correction and for the measurement of scan linearity.

There are numerous other pitch structures on the RM that can be made into secondary calibration structures once the SEM or other instrument has been calibrated. Development of secondary calibration structures is especially useful if the instrument in use is expected to contaminate the RM sample structures.

Figure 4 shows the design and the nominal values for the X and Y direction large pitch structures of one of the non-connected RM 8820 patterns. The two sets of patterns are designed to be identical in both of the X and Y pitch directions. These are marked with numbers 1 and 3 for the X direction and 2 and 4 for the Y direction. All structures were designed to be either parallel with or perpendicular to each other. Between the 50 µm pitch patterns are much smaller pitch patterns, shown at the right side of the drawing of Figure 4.

The finest calibration structures are the 200 nm pitch structures found in the center of the 4 µm pitch structures (schematically shown on the right side of Figure 4). Lithographically they have merged into one larger structure. This has occurred because the resolution of the optical lithography technique for that area was not sufficient to resolve these small pitch structures as binary mask features. The structures marked A to G on Figure 5 were designed to solve this problem and provide 200 nm to 2 µm pitches. The A 200 nm and B 280 nm pitch structures were designed and fabricated with phase- shifting

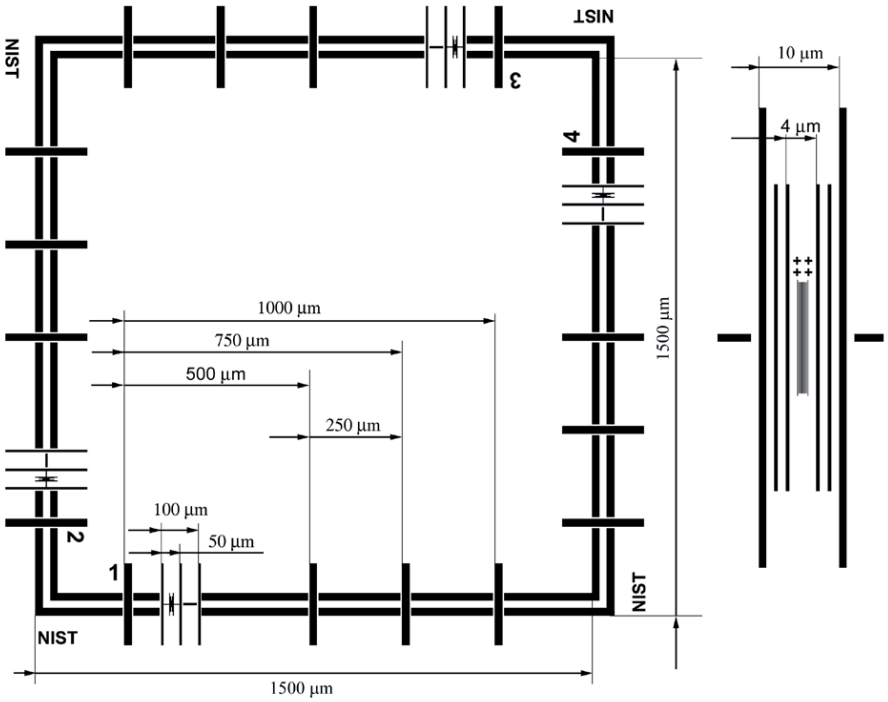

Figure 4. The nominal X and Y direction pitch values for the large structures of RM 8820.

patterns. Therefore, they have been resolved in this location. These are useful for pitch calibration to about 300 000 times magnification. But, as a minor consequence of the phase shifting lithography method, these are not fabricated as equal lines and spaces; the widths of the lines are smaller than the widths of the spaces between them. The C 400 nm, D 500 nm, E 700 nm, F 1 µm and G 2 µm pitch calibration structures were made with the binary mask and were fabricated as relatively equal line widths and space widths. All A to G pitch calibration structures were made as dense lines of large 100 µm by 120 µm areas with the same pitch, and there are dense and isolated lines that extend beyond the dense calibration areas. These were designed to facilitate cross sectional measurements of dense and isolated lines after precision cleaving. All small structures at their bottom also have a 1 µm line, 1 µm space structure and 0, 10, etc. numbers up to 100 to help the identification of exact locations within the small patterns themselves. After locating one of these numbers, one can easily and with very little sample motion arrive at isolated or dense lines or somewhere in the 100 µm by 120 µm areas. This, generally, may be done without any focus adjustment. All patterns with the same designation were designed to be identical, e.g., the four A patterns (two in the X direction and two in the Y direction) are the same design. Figure 5 shows the schematic design of the location of the A through G structures. All other structures have similar designs, but with different pitch values (as described above).

3.3 SEM Imaging. A SEM image of the entire RM 8820 structure, at low magnification, is shown in Figure 6. The pitch values of the large square frame structure are 1.5 mm in both X and Y directions. Figure 7 shows a typical low magnification image of a roughly 50 µm portion of a binary type, 1 µm pitch structure. The 2 µm pitch numbered guidelines at the bottom and the isolated and dense structures are clearly visible. Figure 8 shows a typical SEM image of the 200 nm pitch patterns at 100 000 times magnification.

3.4 Reference Scale Calibration Values. The reference values for a large set of scale calibration values are provided in Table 1. The reference values were determined by combined SEMATECH CD-SEM and laboratory SEM measurements. The reference values supplied are non-certified values that are the best estimate of the true value. These values do not meet all of the NIST criteria for Standard Reference Material certification (at this time), but do provide a reference value. The values are provided with large associated uncertainties since not all of the sources of uncertainty for the Reference Material have been fully explored.

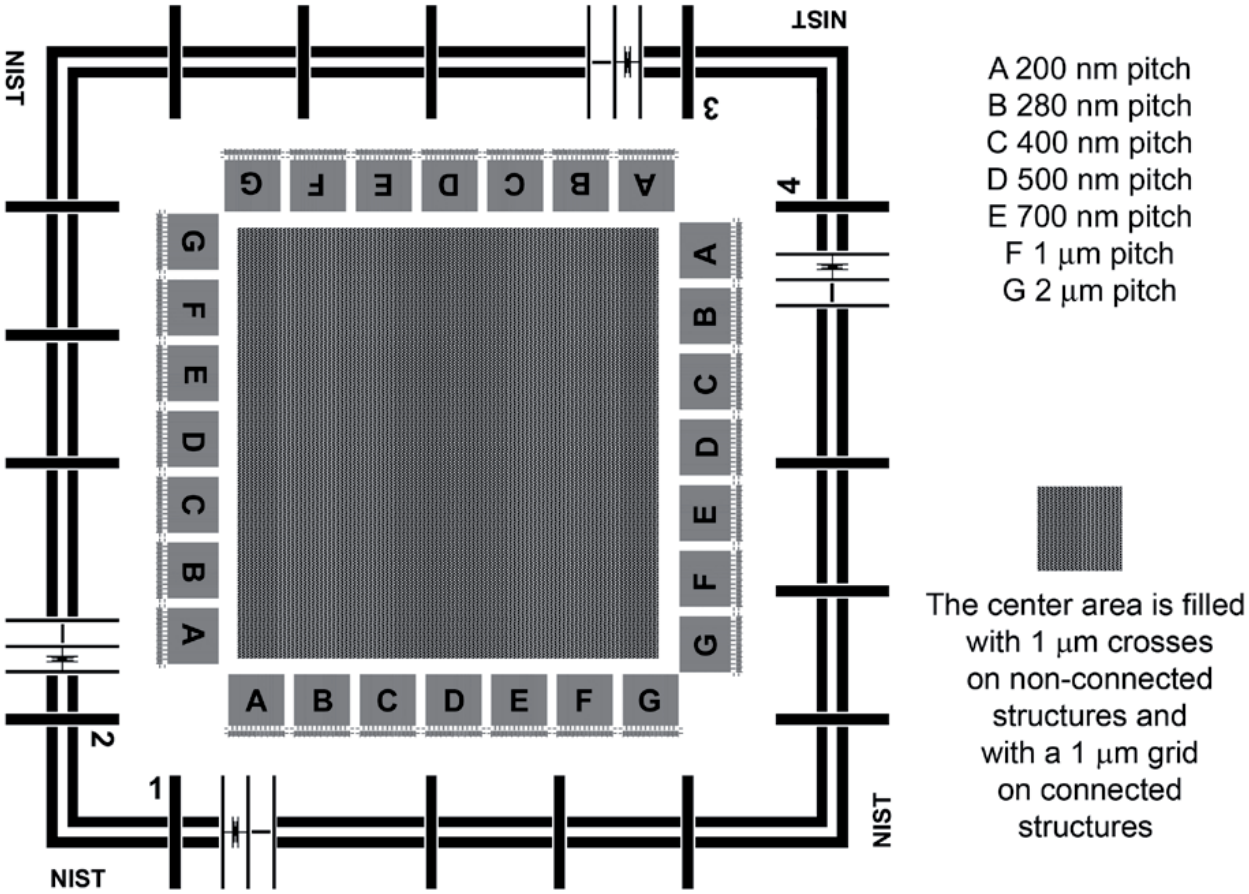

Figure 5. The nominal X and Y direction pitch values for the small A to G structures of RM 8820.

3.5 NIST Pitch Calibration Software Program. A public domain pitch calculation software program is available from NIST which calculates the pitch on hundreds or thousands of locations and gives a statistical measure of the pitch values. The program uses a robust and comprehensive algorithm for measuring pitch in images of calibration samples. The algorithm was implemented and tested in a MATLAB program. The algorithm requires a few parameters to be set before processing: contour level, angular acceptance and confidence level for the periodic pattern matching. However, for most images, the default values found automatically or preset in the program work well. No or little user intervention is therefore needed. This software program was developed by Martin Oral of NIST and is currently available by contacting Andras Vladar (andras.vladar@nist.gov). This program is provided as an aid to the RM users and is not guaranteed to function on all operating systems, but only on those on which it has been tested.

3.6 Contamination-Free Measurements. Electron beam-induced sample contamination is a serious but avoidable problem associated with SEM imaging and metrology. Even a thin carbonaceous layer can significantly and unacceptably alter the sample. The sample surface is never completely clean, because typically a thin layer of molecules containing water and carbon dioxide is on the surface of samples that have been exposed to room air. Other possible contaminant molecules could come from storage containers or from the fabrication processes, the vacuum in the specimen chamber or outgassing from various parts of the sample stage such as any species that have high mobility and long mean free paths in vacuum conditions. Any or all of these contamination sources can end up in the vicinity of the region hit by the electron beam. In a dynamic process of adsorption and desorption, these molecules can be deposited on the surface of the sample. The number of these molecules can be very large that they literally makes various sample features grow. Beyond this, the contamination process changes the very top surface of the original sample to a carbonaceous-like material, which generally leads to a significant drop in the secondary electron emission. Both of these phenomena have detrimental effects: the sample changes its

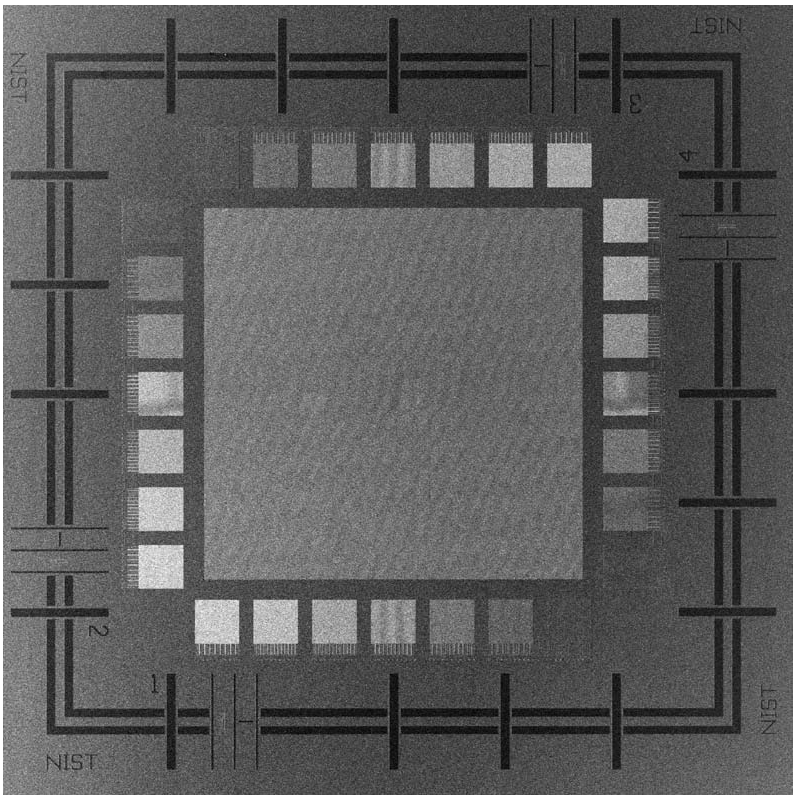

Figure 6. SEM micrograph of the whole RM 8820 structure. The pitch values of the large square frame structure are 1.5 mm in both X and Y directions as shown in Figure 4.

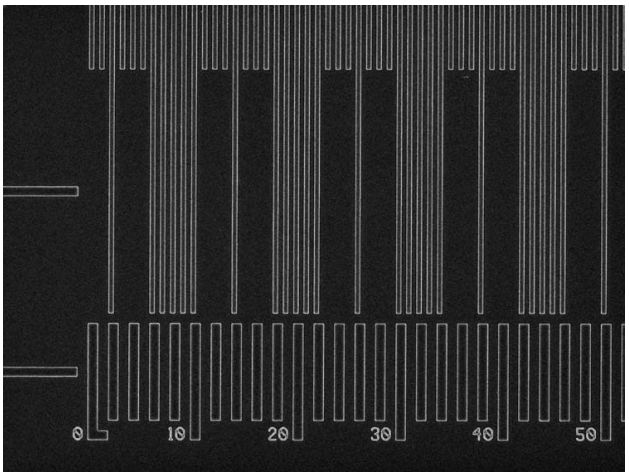

Figure 7. Typical low-magnification image of a roughly 50 µm portion of the binary-type pitch structure. The 2 µm pitch-numbered guiding lines at the bottom and the isolated and dense structures are clearly visible.

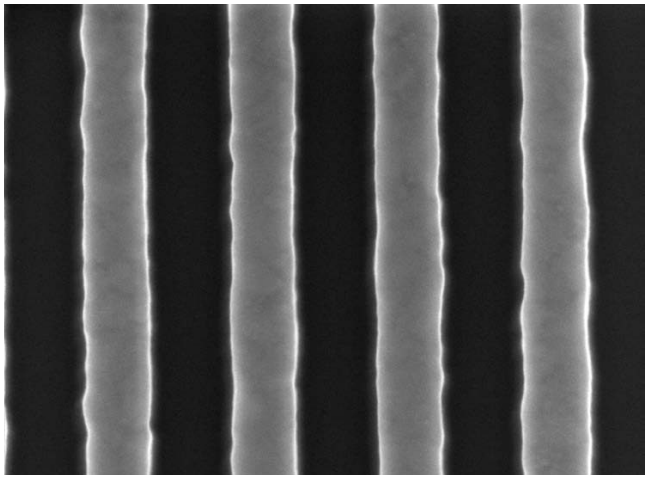

Figure 8. Typical SEM image of the 200 nm pitch patterns at 100 000 times magnification.

RM 8820 Measurements			
Pitches	**Nominal (µm)**	**Metrology Instrument (µm)**	**Inspection Instrument (nm)**
1	**1500**	**1500.1**	**n/a**
2	**1000**	**1000.1**	**n/a**
3	**500**	**500.2**	**n/a**
4	**250**	**250.3**	**n/a**
5	**100**	**100.2**	**n/a**
6	**50**	**50.1**	**n/a**
7	**10**	**10.0**	**n/a**
8	**4**	**4.01**	**n/a**
9	**1.4**	**1.4**	**n/a**
10	**2**	**2.0**	**n/a**
11	**1**	**1.0**	**n/a**
12	**0.7**	**0.70**	**n/a**
13	**0.5**	**0.50**	**n/a**
14	**0.4**	**0.40**	**400.7 STD 1.2**
15	**0.28**	**0.28**	**n/a**
16	**0.2**	**0.20**	**199.6 STD 0.9**

size, which is a critical parameter for IC process control, and the smaller electron emission leads to unpredictable results, because the measurement is reporting results not only on the sample but on the sample with an uncharacterized layer of different material on the top of it. For accurate dimensional measurements and adequate process control, it is essential to minimize electron beam-induced contamination.

Regular monitoring of the contamination performance of the SEM is critical. To separate sample-related and instrument-related contamination and thus avoid misleading results, it is necessary to have a clean sample or a sample that can be cleaned. It was found that amorphous silicon structures on 2 nm gate oxide on a silicon substrate work well for testing charged particle-induced contamination. An acidic, oxidizing, wet chemical solution and thorough rinsing were found to regenerate their cleanliness when necessary [11, 12].

If the instrument fails the cleanliness test with a clean sample, it needs to undergo cleaning. A low-energy oxygen plasma cleaning process has been used successfully [12]. It very effectively eliminates oily residues from the vacuum and from the surfaces within the sample chamber, including the surfaces of the sample stage. It is important to point out that the ionized oxygen generated by the plasma cleaner oxidizes many materials, but, advantageously, the process is especially effective on hydrocarbon residues. It is recommended to use the minimum, but sufficient time and plasma power (5 W – 7 W). Manufacturer recommendations for the operation of the plasma cleaner should be closely followed.

Unnecessary cleaning is not recommended. If the instrument meets the contamination specification, there is no need for cleaning. It takes some time to go through the cleaning process, which involves leaking in air and keeping the sample chamber at around 60 Pa pressure for the time of the plasma cleaning and then pumping it down. Excessive exposure of the sample chamber and sample stage to oxygen plasma might result in undesirable consequences. Again, refer to the manufacturer's recommendations.

4.0 Conclusion

The newly released RM 8820 was developed under the strong collaboration between the NIST Manufacturing Engineering Laboratory and SEMATECH. This standard was designed through the Advanced Metrology Advisory Group and was fabricated at SEMATECH using current advanced lithography techniques. It was designed to

be as versatile as possible and to utilize all the available the real estate on the silicon chip as efficiently as possible. Advanced lithography techniques continue to improve and the next step is a version based upon the AMAG 6 reticle. It is anticipated that this artifact will be released in both an RM and an SRM form some time in the near future.

5.0 Acknowledgements

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6.0 References

[1] Certain commercial equipment is identified in this work to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment identified is necessarily the best available for the purpose.

[2] Contribution of the National Institute of Standards and Technology; not subject to copyright.

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