DEPARTMENT OF COMMERCE • UNITED STATES OF AMERICA

National Institute of Standards & Technology

Report of Investigation

Reference Material 8820

Scanning Electron Microscope Scale Calibration Artifact

Reference Material (RM) 8820 is primarily intended to be used for X and Y scale (or magnification) calibrations from less than 10 times magnifications to more than 100 000 times magnifications in scanning electron microscopes (SEMs). It was designed to provide good contrast at low and high electron landing energies (accelerating voltages). Beyond testing scale calibration, it can be used for non-linearity measurements, especially at lower than 10 000 times magnifications. It can also be used for optical and scanning probe and other types of microscopes. Most SEMs require a set of calibration structures of different sizes to cover the full range of possible magnifications. This Reference Material (in part using the ideas implemented in earlier NIST scale calibration artifacts) is designed to meet that need [1–4]. A unit of RM 8820 consists of a 20 mm × 20 mm lithographically patterned silicon chip.

Scale Calibration Values: The measured values for a large set of pitch spacings are provided in Table 1 as Reference Values and associated uncertainties. These values were determined by combined critical dimension (CD) SEM and laboratory SEM measurements. Reference values are provided with associated uncertainties as a preliminary characterization of the material. They do not meet the NIST criteria for Certified Values because they are not presented with complete uncertainties, the traceability to NIST's realization of the meter is not fully optimized, and the measurement uncertainties are not fully evaluated according to the ISO Guide [5].

RM 8820 is intended to preliminarily address users' needs for an SEM magnification calibration reference material. NIST is continuing to work on improving the characterization of this RM to upgrade the quality of the measured values. Registration (see attached sheet) will facilitate notification of such improvements.

Expiration of Value Assignment: The Reference Values in Table 1 are valid indefinitely provided the RM is handled and stored in accordance with instructions given in this Report (see "Handling and Storage"), and the RM is not damaged, contaminated, or otherwise modified. Periodic recalibration of this RM is not required.

Handling and Storage: Care must be exercised when handling this RM. Avoid touching the surface with the microscope objective lens while setting up or focusing. Only non-abrasive and non-corrosive cleaning techniques should be used, as abrasives and acids can alter the line edges. When not in use, RM 8820 should be stored in the case supplied or equivalent and kept at room temperature.

The technical direction for this reference material was provided by A.E. Vladar and M.T. Postek of the NIST Precision Engineering Division, and by M. Bishop of International SEMATECH.

The coordination of the investigation of this RM was performed by A.E. Vladar of the NIST Precision Engineering Division. The reference values in Table 1 were provided by A.E. Vladar of the NIST Precision Engineering Division.

Statistical consultation leading to the reference values was performed by N.F. Zhang of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this RM were coordinated through the NIST Measurement Services Division.

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RM 8820 has been fabricated on a 20 mm × 20 mm silicon chip as parts of a large set of structures that was designed for various dimensional metrology purposes for semiconductor technologies as seen in Figure 1.

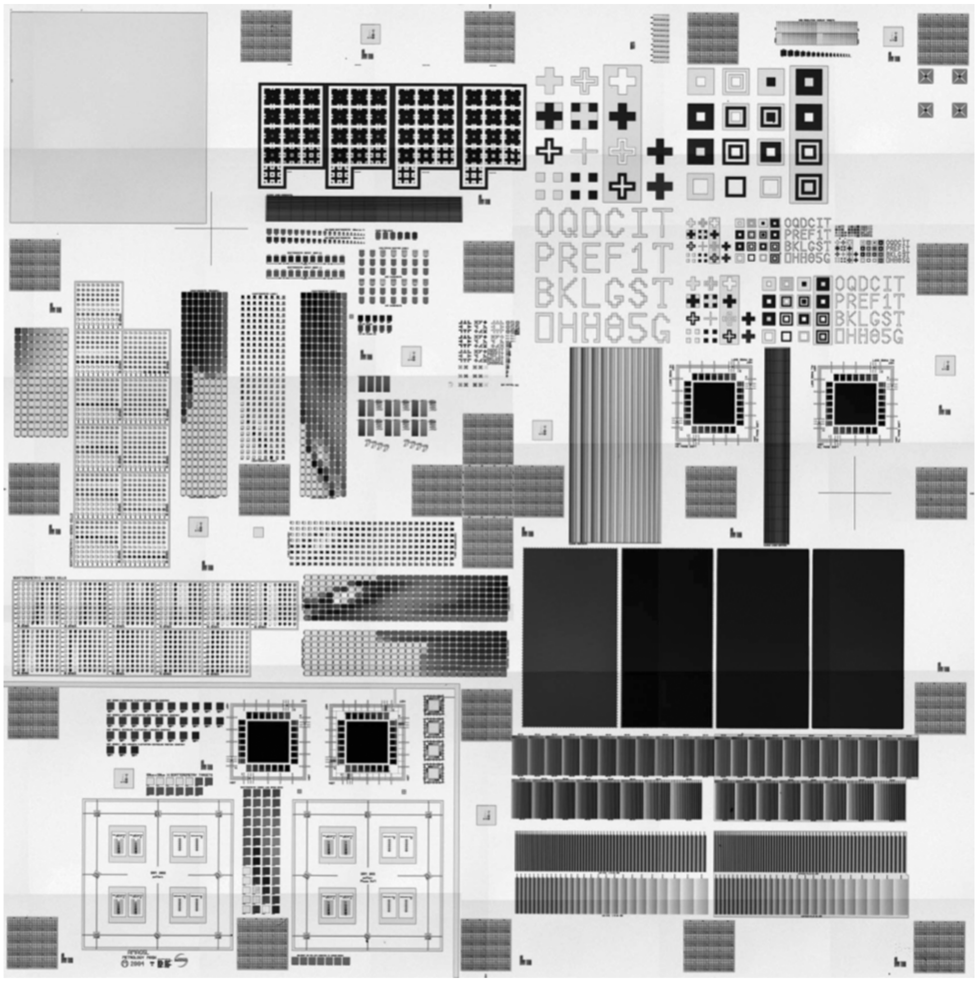

Figure 1. Optical microscope view of the 20 mm × 20 mm chip.

The RM 8820 parts of these structures are marked with letters “NIST” and are readily visible with the naked eye as small bright squares within the large chip. These structures have pitches ranging from 200 nm to 1.5 mm in both X and Y directions (Figure 2).

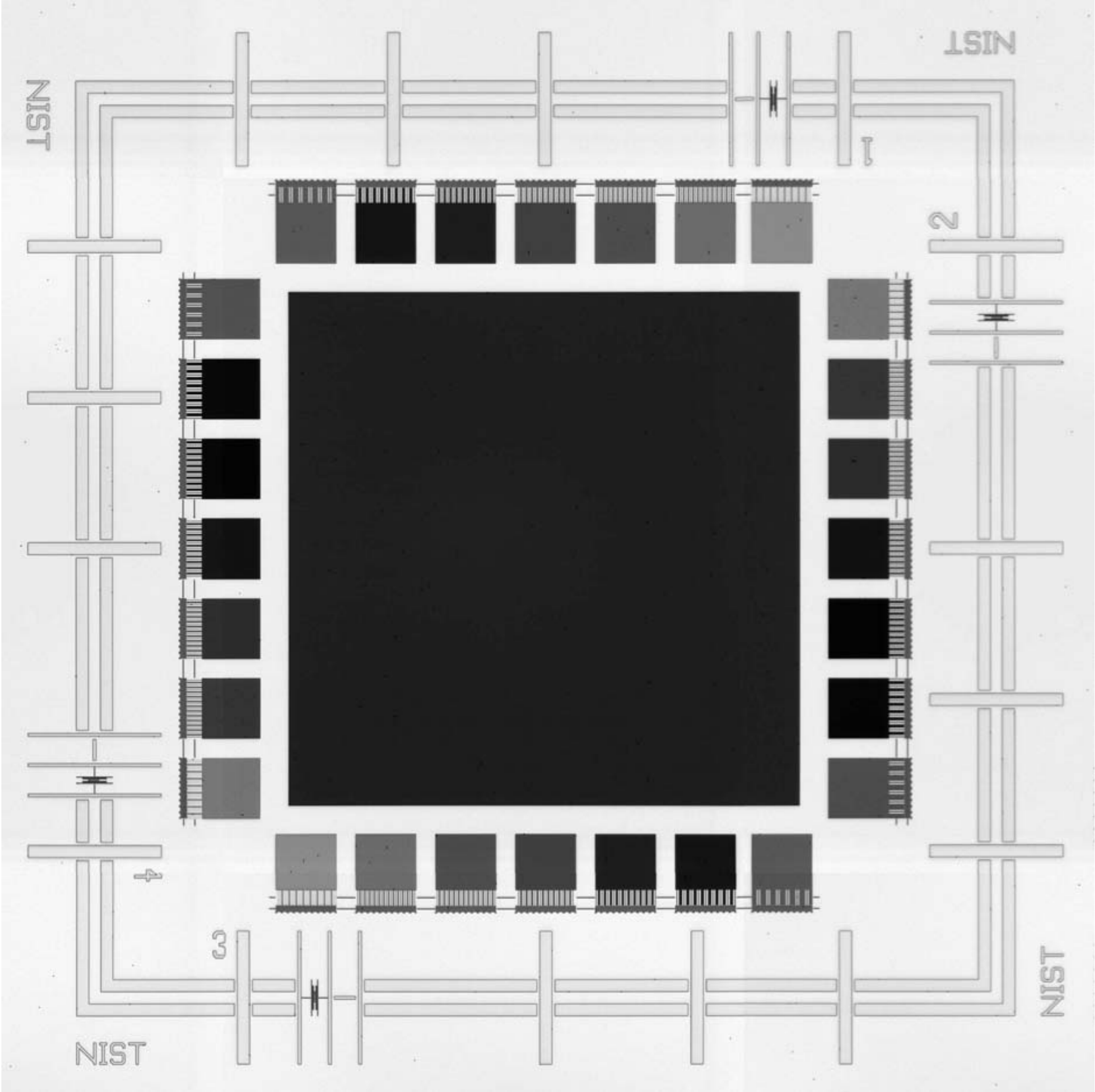

Figure 2. Optical microscope view of the 1.5 mm × 1.5 mm RM 8820 test structure.

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

Figure 3 shows the design and the nominal values for the X and Y direction large-pitch structures of RM 8820. There are two sets of designed-to-be identical X and Y pitch calibration structures. 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, there are further smaller pitch patterns shown at the right side of the drawing in Figure 3.

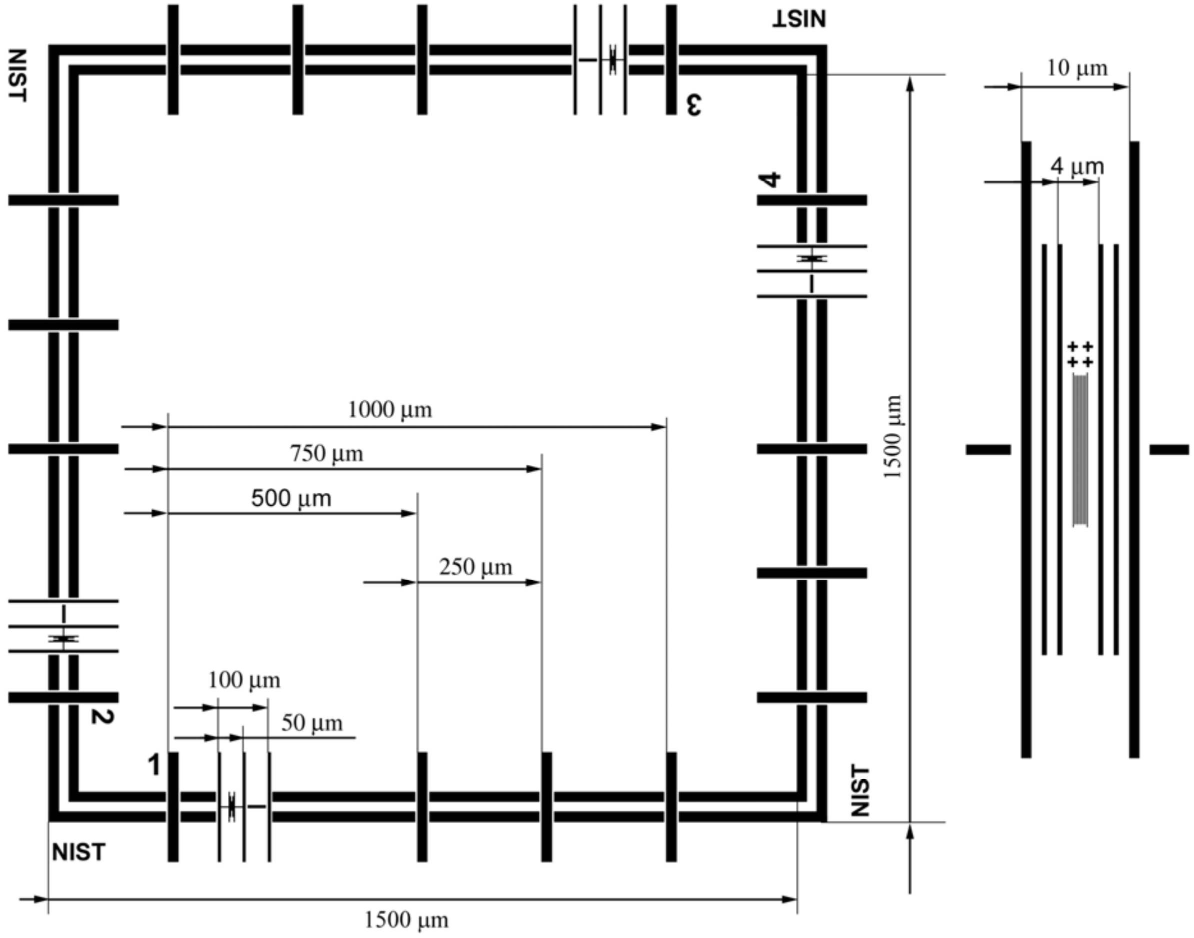

Figure 3. The nominal X and Y direction pitch values for the large structures of RM 8820.

The 200 nm pitch structures that are in the centers of the 10 µm and 4 µm pitch structures (schematically shown on the right side of Figure 3) are merged into one larger structure. This has occurred because the resolution of the lithography technique was not sufficient to print these small pitch structures as binary mask features. The 100 µm × 120 µm areas marked A through G on Figure 4 were designed to provide 200 nm to 2 µm pitches. The A 200 nm and B 280 nm pitch structures were designed and fabricated with phase shifting patterns. Therefore, they have been resolved. These are made to be useful for pitch calibration, but as a consequence of the phase shifting lithography method, 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 as binary mask structures and are formed with close to equal line widths and space widths. All A to G pitch calibration structures were made as dense lines of large (100 µm × 120 µm) areas with the same pitch, and there are dense and isolated lines that extend beyond dense calibration areas. These were designed to facilitate cross-sectional measurements of dense and isolated lines after precision cleaving. All A to G pitch calibration structures at their bottom 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 × 120 µm areas, probably without needing 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).

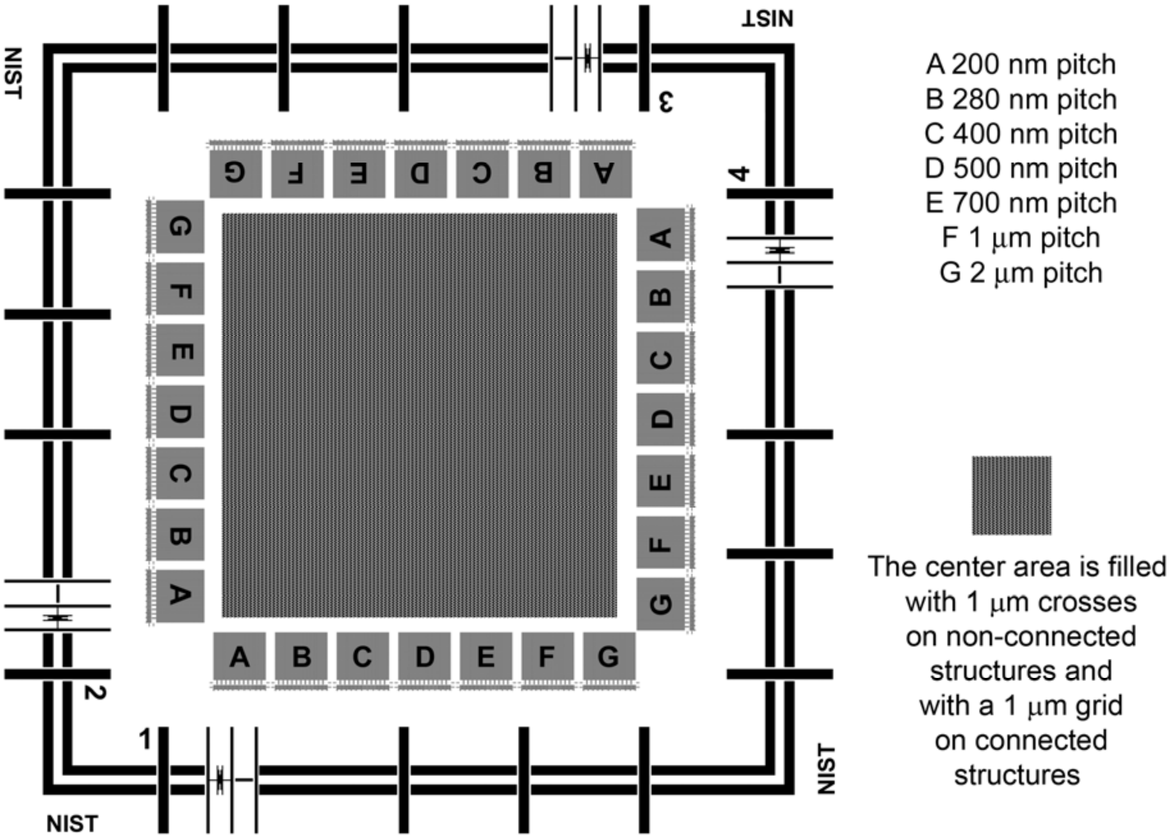

Figure 4. The nominal X and Y direction pitch values for the small A to G structures of RM 8820.

Figure 5 shows the schematic design of structures A and G. All other structures have similar designs, but with different pitch values.

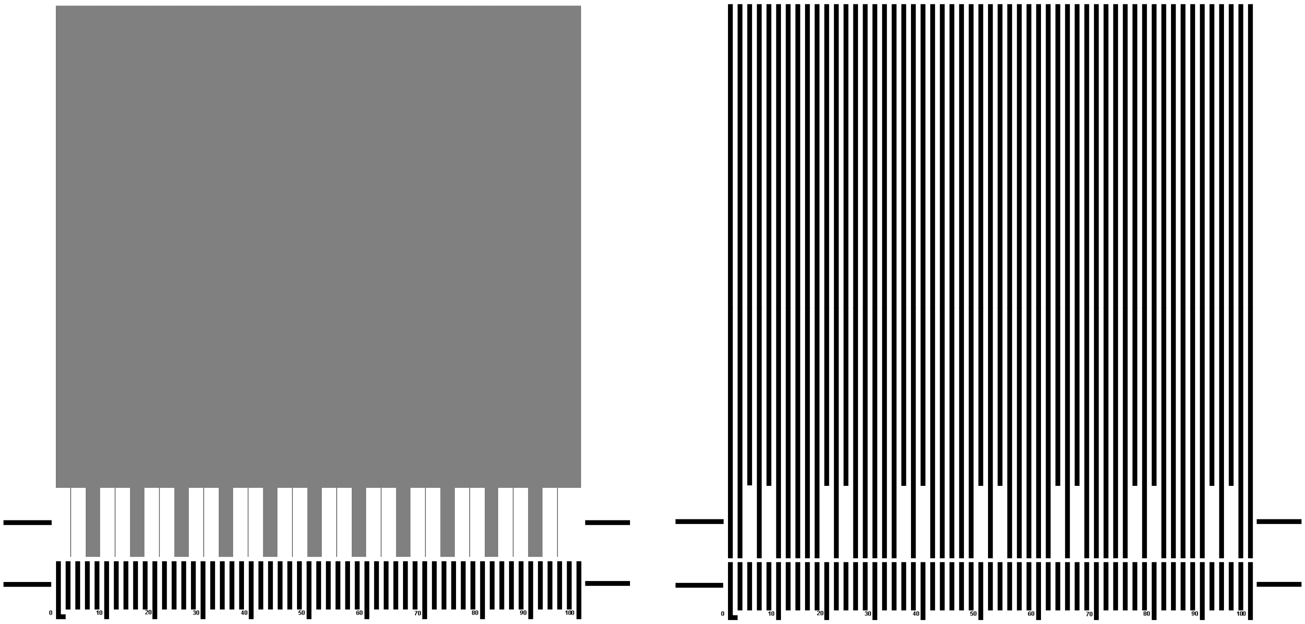

Figure 5. Schematic design of structures A (left) and G (right).

The SEM image of RM 8820 is shown on Figure 6. Figure 7 shows the center cross and grid patterns.

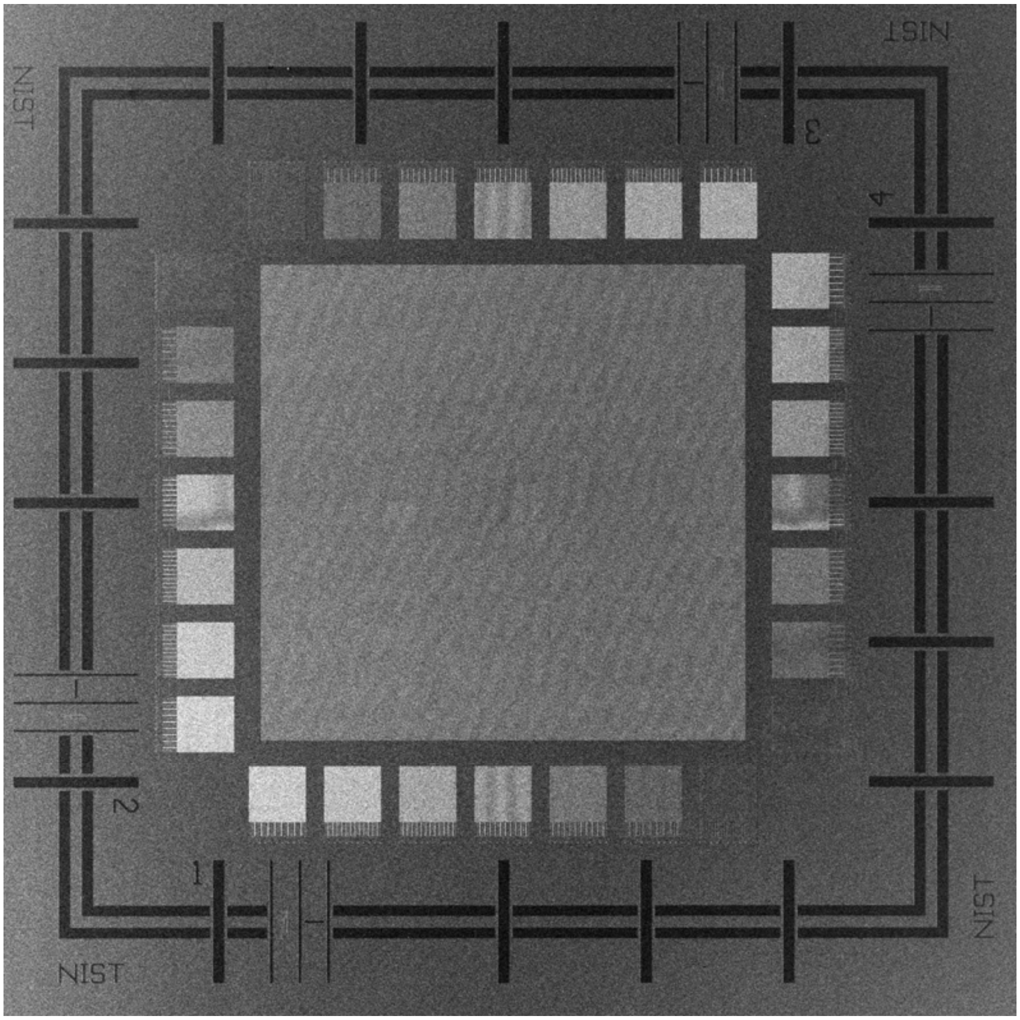

Figure 6. The SEM image of the whole RM 8820 structure. The pitch values of the large square frame structure are 1.5 mm in both X and Y directions as shown in Figure 3.

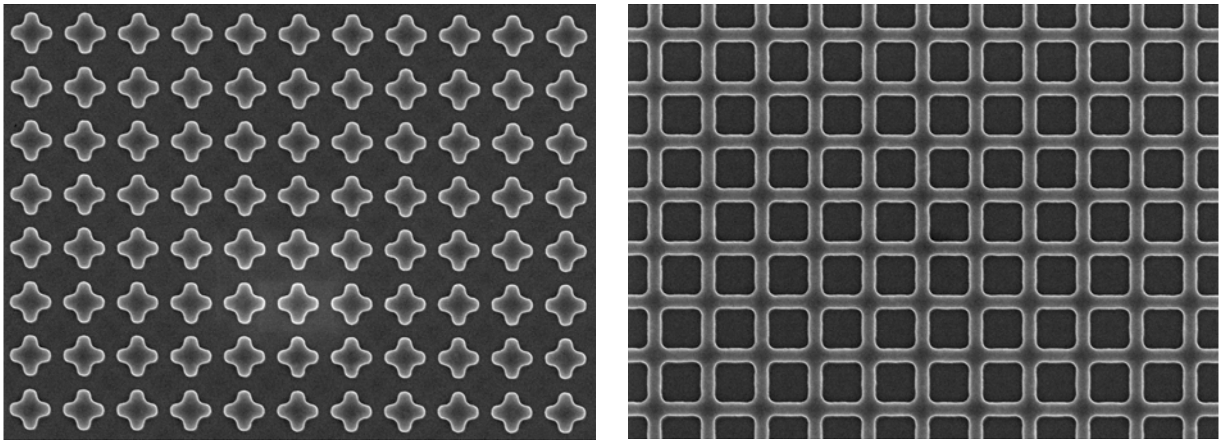

Figure 7. The SEM images of the center cross (left) and grid (right) structures of RM 8820. The pitch values of the structures are 1 μm in both X and Y directions.

Figure 8 shows a 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 9 shows the similar, but 200 nm pitch, phase-shifting structures.

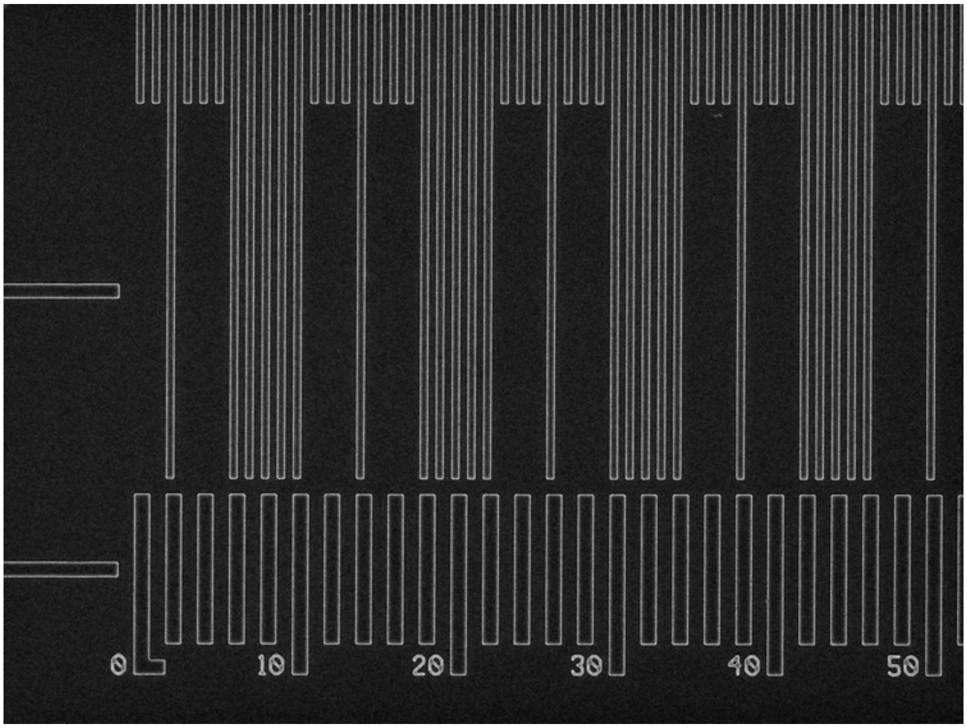

Figure 8. Typical low-magnification SEM image of a roughly 50 μm portion of binary-type pitch structure.

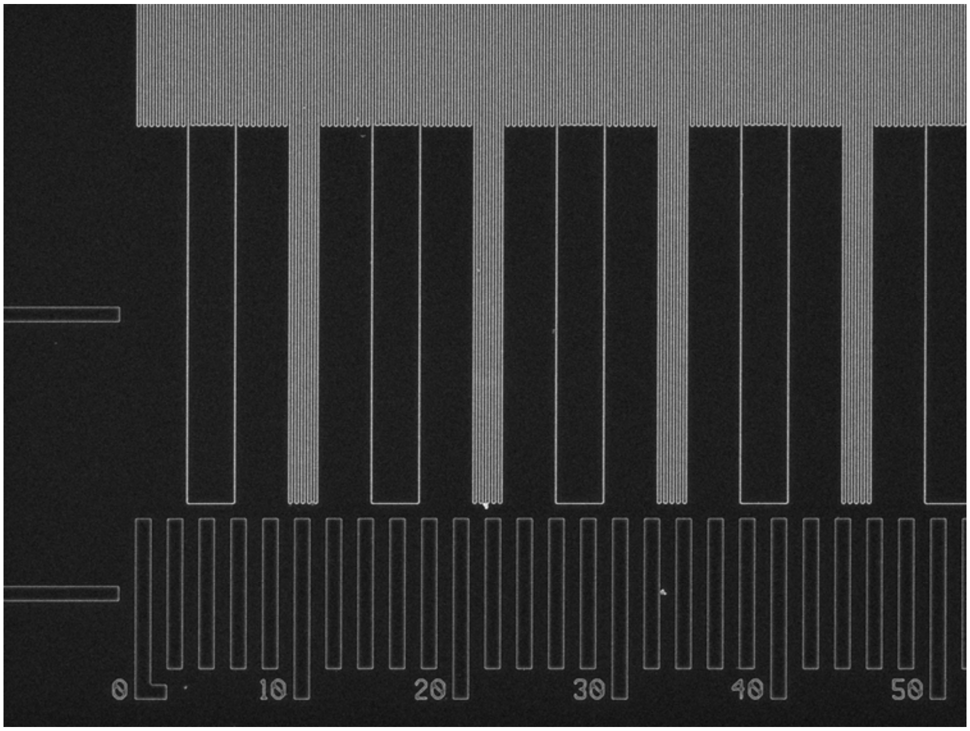

Figure 9. Typical low-magnification SEM image of the 200 nm pitch phase shifting structures.

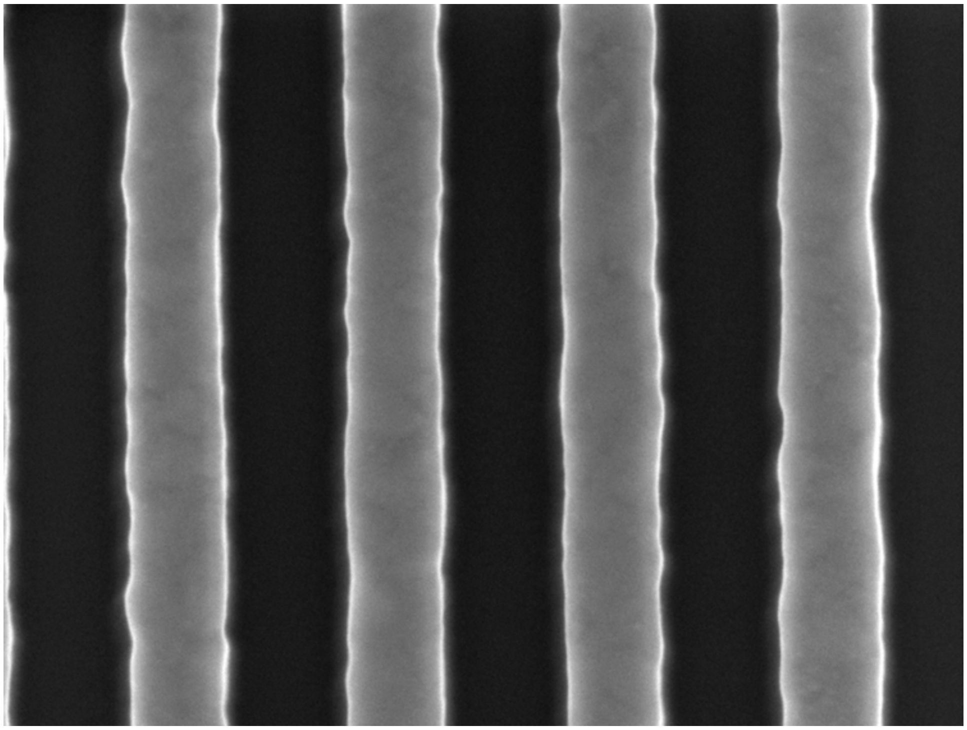

Figure 10. Typical SEM image of the 200 nm pitch patterns at 100 000 times magnification.

The RM 8820 samples were fabricated on 200 mm 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 (SiO_2) layer in between them. This 2 nm thin SiO_2 was used as an etch stop [6–9]. Before the patterns were made, optical film thickness 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 (one standard deviation = 1.6 nm). All amorphous silicon patterns have edge unevenness or edge roughness, which is the result of the lithography and etch processes used in the fabrication of RM 8820. This edge roughness, however, does not have a very large effect on the pitch determination if a large enough number of pitches are considered in each pitch measurement. This can be achieved by using the center two-thirds of the images taken at suitable magnifications.

Each sample is provided in a box lined with semi-sticky plastic to protect the sample from damage. It is important to carefully use (preferably conductive plastic) tools to remove the chip from its box. The semi-sticky plastic will allow the user to 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.

The chip is to be mounted by the user on the proper stub, wafer, or sample holder suitable for his/her particular instrument. Utmost care should be taken in the handling and mounting of the sample. Spring-loaded fasteners or very small amounts of carbon-conductive paste applied at two corners of the chip were found to work well. Electron beam-induced contamination might 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, but it is possible to achieve cleanliness so that even after 10 minutes of continuous imaging there will be no visible change in the quality of the image and the measured value of the pitch. This may require the use of effective methods that are designed to clean the sample and the sample chamber and the vacuum system. If sample cleaning becomes necessary, it is recommended to study and take advantage of the information of pertinent literature. Chemical and/or low-energy plasma cleaning were found to be useful, but caution must be exercised as both these and other treatments may ruin the sample.

Measurements of 64 scans were averaged for the Metrology Instrument (critical dimension measurement SEM, CD-SEM) results provided in Table 1. The Inspection Instrument (NIST Laboratory SEM, FEI Helios SEM[1]) used an average of hundreds of lines.

Table 1. Nominal and measured Reference pitch values for RM 8820

Pitches	Nominal Value (µm)	Measured Pitch Spacings[(a)] Value (µm)	Uncertainty k=2 (µm)	CD-SEM Measured Values Value (nm)	Uncertainty (nm)
1	1500	1500	92.6	n/a	
2	1000	1002	62	n/a	
3	500	501.2	30.9	n/a	
4	250	251.2	15.5	n/a	
5	100	100.4	6.23	n/a	
6	50	50.2	3.16	n/a	
7	10	10.05	0.63	n/a	
8	4	4.018	0.25	n/a	
10	2	2.010	0.13	n/a	
11	1	1.005	0.06	n/a	
12	0.7	0.704	0.04	n/a	
13	0.5	0.503	0.03	n/a	
14	0.4	0.402	0.02	400.7	$s = 1.2$[(b)]
15	0.28	0.282	0.02	n/a	
16	0.2	0.201	0.01	199.6	$s = 0.9$[(b)]

[(a)] The Measured Reference values are provided with associated uncertainties as a preliminary characterization of the material. They do not meet the NIST criteria for Certified Values because they are not presented with complete uncertainties, the traceability to NIST's realization of the meter is not fully optimized, and the measurement uncertainties are not fully evaluated according to the ISO Guide [5].

[(b)] The value s is the standard deviation of the repeatability of multiple CD-SEM measurements.

RM 8820 was produced at International SEMATECH (Austin, TX). An Applied Materials CD-SEM equipped with a thermally assisted field emission source was used at International SEMATECH for the automatic measurements of the samples in their 200 mm wafer state. After dicing, several chips were inspected for quality and for the pitch values in the NIST Hitachi S-4800 and FEI Helios SEMs.

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[1] Certain commercial equipment, instruments or materials are identified in this report to adequately specify 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 materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

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