NBSIR 87-3665

Low Accelerating Voltage Pitch Standard Based on the Modification of NBS SRM 484

Michael T. Postek

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Microelectronics Dimensional Metrology
Technology A-347
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U.S. DEPARTMENT OF COMMERCE, C. William Verity, *Acting Secretary*
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Preface

This work was conducted as part of the Microelectronics Dimensional Metrology program at the National Bureau of Standards (NBS). The portion of the work described in this report was partially supported by the Harry Diamond Laboratories, Adelphi, Maryland under MIPR #R86-0086. The contract was monitored by Mr. Robert Reams of HDL and the point of contact at NBS for information about the technical elements of this project is Dr. Michael. T. Postek of the Microelectronics Dimensional Metrology Group at the National Bureau of Standards, Gaithersburg, Maryland.

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ABSTRACT

The National Bureau of Standards is actively developing micrometer and submicrometer standards for the scanning electron microscope. This report summarizes the progress made to extend the imaging range of the presently available SRM 484 for use as an interim standard for low accelerating voltage magnification calibration applications for this instrument.

INTRODUCTION

The National Bureau of Standards is actively developing micrometer and submicrometer standards for dimensional metrology in the scanning electron microscope (SEM). The only magnification standard reference material (SRM) presently available for calibrating scanning electron microscopes is SRM 484.[1,2] This standard provides a known pitch between gold lines in a nickel matrix and has proven useful for many SEM applications. However, SRM 484 was developed prior to the recent interest in nondestructive, low accelerating voltage SEM operation. The driving force for nondestructive inspection at low accelerating voltages has been the semiconductor industry for integrated circuit mask and wafer inspection and measurement. In its present form, SRM 484 is unsuitable for use in new nondestructive inspection instruments for two main reasons: a lack of suitable contrast below about the 5.0 keV accelerating voltage range and the overall size which is not directly compatible (due to the thickness which greatly exceeds that of a semiconductor wafer or mask) with newly introduced dedicated SEM inspection instrumentation.[3] A project was initiated in FY 1986 at NBS, co-sponsored by Harry Diamond Laboratories, to physically

modify SRM 484 to make it suitable for use at low accelerating voltage operation without altering its calibration or certification procedures.

A sample capable of being used to calibrate the magnification of an SEM at low accelerating voltage (e.g., 1.0 keV or less) is a prerequisite for precise and ultimately accurate measurements made in this instrument. Correct adjustment of the magnification to a known standard is primary to this operation because the measurement information is obtained directly from the size of the scan (magnification) as related to final lens voltage. Present magnification calibration procedures are done only at high accelerating voltages [1] and the correct magnification information is expected to be automatically compensated for in the instrument as the accelerating voltage is lowered. This may not be the case since unpredictable instrument factors may come into play (e.g., lens hysteresis), factors that will invalidate the automatic compensation expectation. Presently, there is no way to check for these effects at the lower accelerating voltages with a recognized standard.

This work is the initial phase of a larger NBS program to develop suitable submicrometer standards for scanning electron microscope linewidth and feature-size measurements. The linewidth measurement standards developed for the optical microscope, SRM 473, 474, and 475 are not designed, or recommended, for linewidth calibration use in the SEM, and they should not be used for this purpose.[4] The optical theory and modelling used in the certification of these optical microscope linewidth SRMs are not directly adaptable to the SEM and, therefore, the criteria developed to determine the edge location are not applicable for anything but an optical measurement. The electron beam effects and the requirements for computer modelling in the SEM are totally different from the diffraction effects in the optical microscope.[4] SRM 474 could, however, be used to calibrate the magnification at low accelerating voltage in an SEM under conditions where the SRM is not charging. In this mode, the magnification of the instrument could be calibrated with the pitch patterns on the SRM. However, continuing this calibration process to include linewidth measurements is not recommended because such calibration results would be only valid for identical chrome-on-glass photomasks. There is no advantage to use the SRM 474 for SEM pitch calibration (except for its smaller size) since this standard is rather expensive relative to SRM 484 and because of potential charging of its glass substrate and electrically isolated metal lines. Coating the sample with a conductive material such as carbon to reduce the possibility of charging would destroy the certification of SRM for optical microscopy. Therefore, a need presently exists for an inexpensive low accelerating voltage magnification standard for SEM calibration.

This report summarizes the progress made to extend the imaging range of the presently available SRM 484 for use as an interim standard for low accelerating voltage applications.

MATERIALS AND METHODS

Several SRM 484 samples were obtained from the Office of Standard Reference Materials (OSRM) at NBS prior to their certification. These samples underwent varying experiments to determine the proper procedures for improving the contrast at low accelerating voltages.

Producing Topographic Contrast by Sample Etching

Sputter Etching. Samples of SRM 484 obtained from the NBS Office of Standard Reference Materials were tested with various sputter etching techniques. Sputter etching is recommended as a procedure to improve the contrast of this sample for optical microscopy.[2] The sputter etching, in this case, is designed to etch the gold lines below the level of the nickel matrix and thereby produce sufficient topographic contrast to improve the image of the SRM at low accelerating voltages. It was initially thought that this would be the simplest and most effective modifying technique. Unfortunately, the sputter etching did not produce sufficient contrast at low accelerating voltage to image the certification lines.

Plasma Etching. The plasma etching technique was considered, but no suitable system capable of etching the nickel could be found.

Wet Chemical Etching. Wet chemical etching was also evaluated. Several of the above mentioned samples were tested in nickel etches.[5] The best of those tested which resulted in acceptable contrast at low accelerating voltages was the following:

Etchant for Nickel/Iron Alloys [5]
5.0g. Ferric Chloride
15ml Hydrochloric Acid
60ml Methyl alcohol

Etching Apparatus. A simple etching apparatus consisting of a wide mouth dish holding approximately 700 ml, dessicator shelf and magnetic stirrer was used for the etching. The sample was held over the magnetic stirring bar by the ceramic dessicator shelf while being immersed in the circulating etching solution. The magnetic stirrer ensured that fresh etchant bathed the sample at all times.

Etching Procedure. The following procedure for the etching was experimentally determined and was done at room temperature (20°C):

1) Prepare the etching solution fresh prior to the procedure.
2) Set a timer for four minutes.
3) Place the cleaned (see below) sample into the etching solution.
4) Start the timer and the magnetic stirrer.
5) After four minutes remove the sample and immerse it into a petrie dish containing fresh methyl alcohol.
6) Thoroughly rinse the sample with methyl alcohol.
7) Dry the sample with dry nitrogen.

Sample Contamination

One serious problem encountered with the wet chemical etching is an extreme sensitivity of the reactants to surface contamination from residual hydrocarbons pinned to the sample surface after observation in the SEM. These hydrocarbons are often deposited on the SRM by the electron beam in normal usage during calibration procedures. The level of contamination will vary from sample to sample depending upon the age of the sample and relative amount of use. Surface contamination causes the etchant to preferentially remove material from the contaminated regions more rapidly than non- contaminated areas. Samples previously viewed in the SEM (especially those viewed at high accelerating voltages and high beam currents) have these residual hydrocarbons pinned on the surface in the area near the Knoop indentation line (see figure in Reference 2) where the initial sample certification and subsequent instrument calibration is done. Therefore, samples viewed in the SEM prior to etching (and thereby possibly contaminated) must be cleaned. The level of surface contamination can be ascessed by observing the sample at low accelerating voltage (figs. 1 A&B.). A cleaning procedure for removal of the carbonaceous deposit is outlined in the documentation that comes with each SRM.[2] This procedure involves carefully polishing the sample on a stationary surface covered with micro-cloth, using metallographic polishing powder. Extreme care must be taken in order to not obscure the Knoop indentation mark. If this procedure is carried out properly, the certification should not be compromised. There are commercial companies that will perform this procedure.[6]

TESTING OF THE CONTRAST-ENHANCED PROTOTYPE SRM

One test of the applicability of any standard is the usefulness in a variety of instruments. One prototype sample successfully etched was viewed in several dif-

ferent instruments of various manufacturers. The basic finding was that this sample was useful at all accelerating voltages in those instruments equipped with "pinhole" lenses. Micrographs of the contrast enhanced sample are shown in figures 2 A&B. If this sample was viewed in a "wide-bore" final lens instrument like those presently utilizing through-the-lens secondary electron collection, the sample became magnetized from the magnetic field leakage of the final lens and uncorrectable astigmatism at low accelerating voltage resulted. This proves a fundamental problem with SRM 484 that is not related to the present etching modification (as the same astigmatism problem would also result if the sample was able to be viewed unetched) and suggests that a radically new design for low acceleration voltage calibration purposes is required.

A second complication imposed by the etching procedure is that the once smooth, highly polished surface is roughened (fig. 2). This results in calibration uncertainity of the line position when an etched nickel grain is located near the gold line.

CONCLUSIONS

The low accelerating voltage version of SRM 484 provides a magnification standard that is designed to bridge the gap until a new lithographically produced SRM can be designed and developed. Modified samples have been successfully made, tested and their limitations determined. This modification to increase contrast of SRM 484 by wet chemical etching was successful and the resulting specimen can be used as a magnification standard (if care is taken) in pinhole lens instruments at low accelerating voltage where the final lens field leakage is minimal. However, due to the limitations found, and the problems encountered in the etching procedures a lithographically produced sample is needed to successfully serve as a SEM standard for the semiconductor community.

ACKNOWLEDGEMENTS

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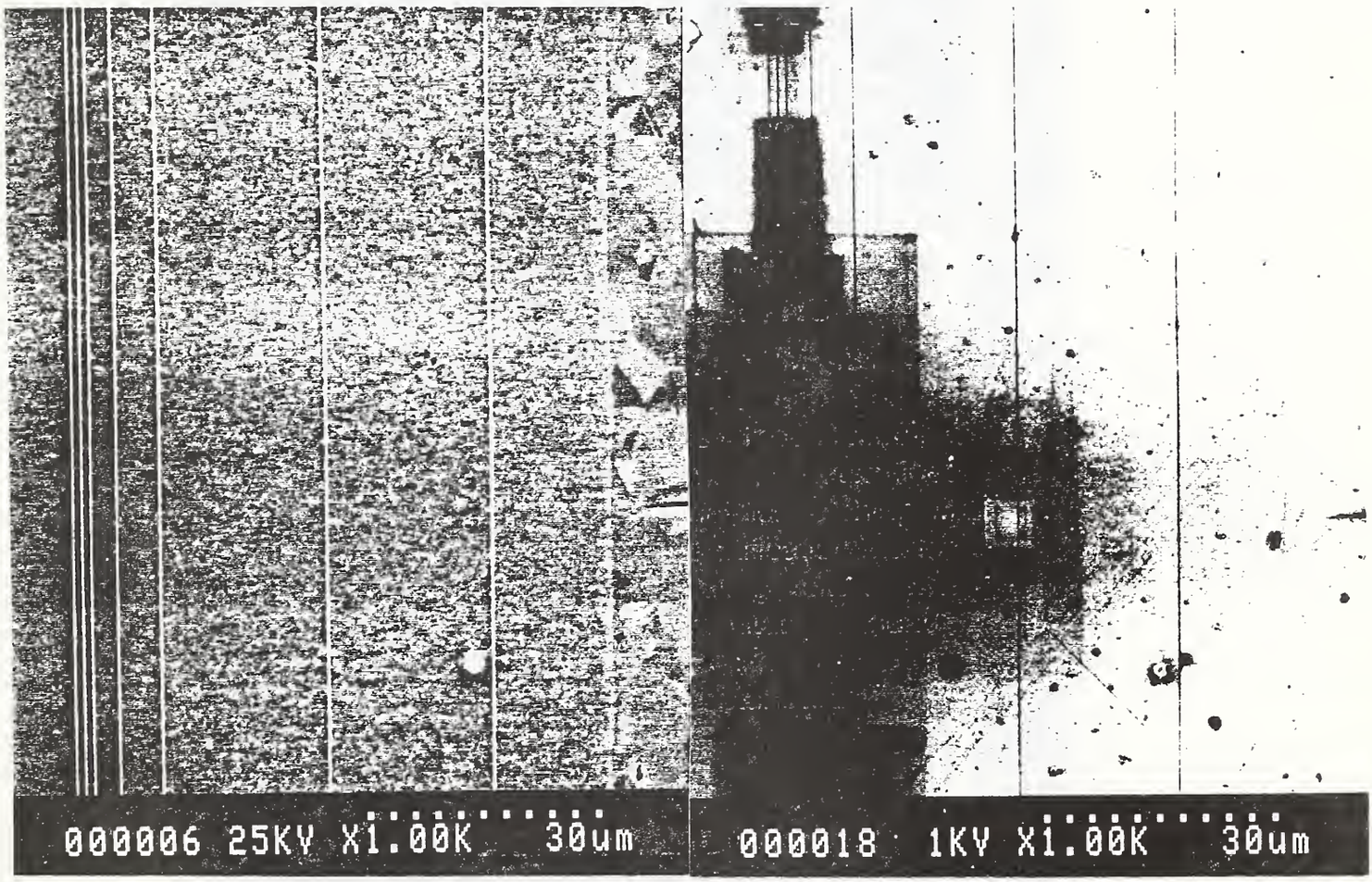

Figure 1. Sample contamination of SRM 484. (A.) Scanning electron micrograph at high accelerating voltage (30keV) of the area adjacent to the Knoop indentation mark of a SRM 484 that has been in use for several years. (B.) Scanning electron micrograph of the same area taken at low accelerating voltage (1.0keV). Note that the arrow points out a common structure.

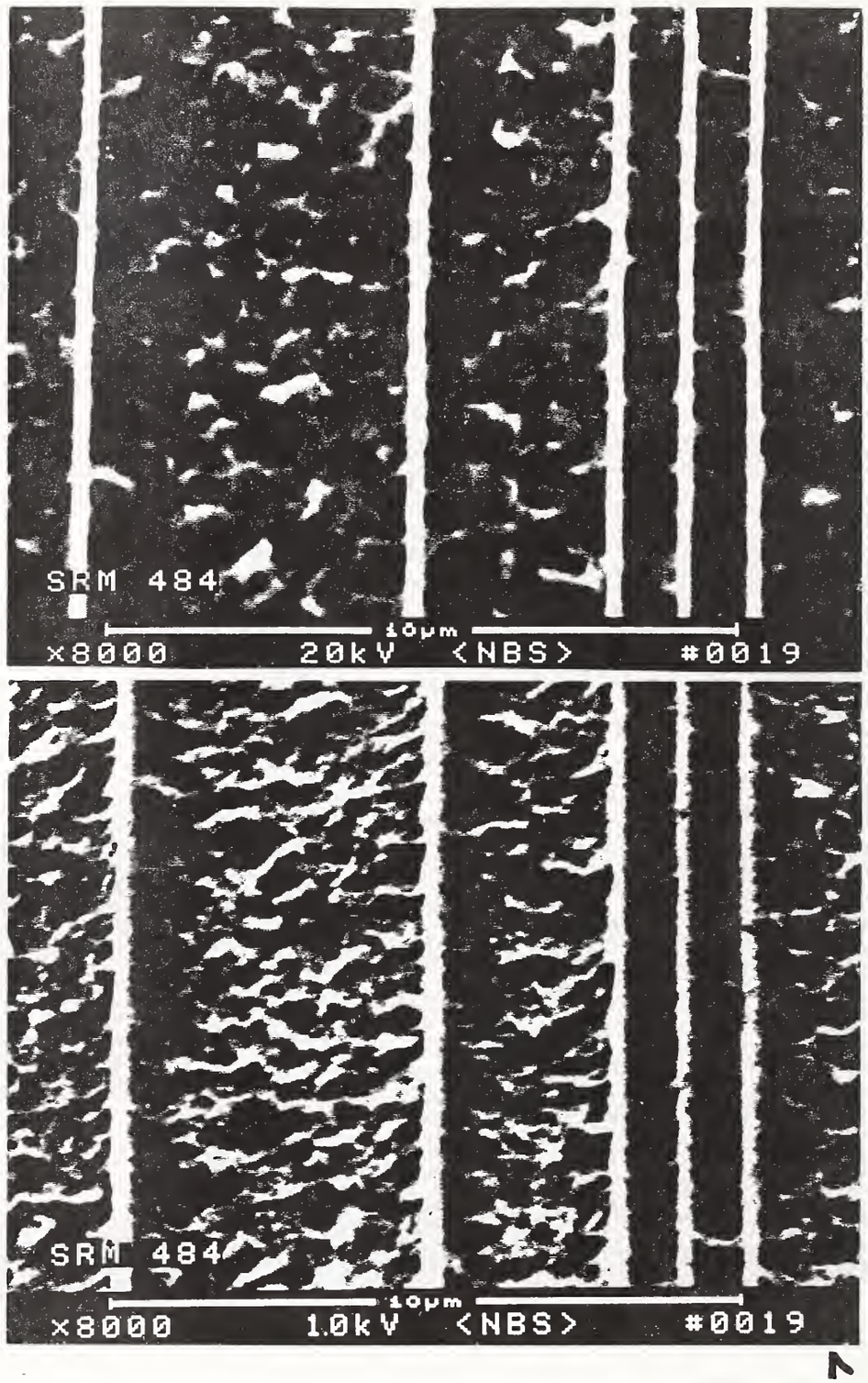

Figure 2. Scanning electron micrographs of the contrast enhanced SRM 484 following the described etching procedure. (A.) High accelerating voltage (20keV). (B.) Low accelerating voltage (1.0keV).

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The National Bureau of Standards is actively developing micrometer and submicrometer standards for the scanning electron microscope. This report summarizes the progress made to extend the imaging range of the presently available SRM 484 for use as an interim standard for low accelerating voltage magnification calibration applications for this instrument.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

etch; low accelerating voltage; scanning electron microscope; SRM; standard

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