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HIGH ENERGY COLLIMATING FINE GRIDS

Final Technical Report

February 1995

Contract NAS5-32530

NASA Goddard Space Flight Center
Greenbelt, MD 20771

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INTRODUCTION AND BACKGROUND

This is the final technical report submitted by Ultramet, 12173 Montague Street, Pacoima, CA 91331 to NASA Goddard Space Flight Center, Greenbelt, MD 20771 under contract NAS5-32530. The period of this contract was from 18 May 1993 to 20 March 1994. The Ultramet project manager was Dr. Robert H. Tuffias, supported by Victor M. Arrieta as project engineer and Dr. Raffaele La Ferla as senior scientist. The NASA Goddard COTR was Charles Katz.

Astronomical observation and study rely on the accurate determination of radiation levels, in order to both observe distant objects and accurately quantify exposure levels. Several upcoming missions will deploy X-ray and gamma-ray imaging systems that image specific wavelengths (or, alternatively, energy levels) of high-energy electromagnetic radiation. To provide accurate imaging systems, collimating grids are required that eliminate off-axis radiation and provide imaging of an extremely small arc angle. A series of collimating grids is envisioned, with each grid pair (grid spacing) designed for the imaging of specific energy levels. Similar to optical, infrared, and radio telescopes, this capability provides imaging and mapping of specific wavelengths of high-energy radiation. Unfortunately, the fabrication techniques investigated previously (mainly electrodischarge machining of slots) have not been able to meet the demanding tolerances (typically 0.0001" or less) required to meet grid performance goals. Because of the high aspect ratios required in the collimating grid slots and the extreme tolerances that must be met, it was not expected that alternate machining techniques (e.g. abrasive grinding, chemical, photochemical, laser, abrasive, water jet) would be able to successfully fabricate the fine grids required.

The objective of this project was to demonstrate the fabrication of extremely tight tolerance collimating grids using a high-Z material, specifically tungsten. The approach taken was to fabricate grids by a replication method involving the coating of a silicon grid substrate with tungsten by chemical vapor deposition (CVD). A negative of the desired grid structure was fabricated in silicon using highly advanced wafering techniques developed for the semiconductor industry and capable of producing the required tolerances. Using diamond wafering blades, a network of accurately spaced slots was machined into a single-crystal silicon surface. These slots were then filled with tungsten by CVD, via the hydrogen reduction of tungsten hexafluoride. Following tungsten deposition, the silicon negative was etched away to leave the tungsten collimating grid structure.

The primary difficulty in this method lies in the ability to completely fill the high aspect ratio slots (approximately 50:1 for 0.1-mm pitch grids) with tungsten. This was accomplished through a directional growth thermal gradient processing technique developed at Ultramet for densification of metal matrix composites. This deposition technique utilizes a thermal gradient across the slot, such that deposit growth is initiated at the bottom of the slot and proceeds to grow vertically to fill the slot. The machined silicon negative grids were supplied by the Jet Propulsion Laboratory (NASA/JPL).

The project was divided into five tasks:

Task 1: Identify materials of construction for the replica and final collimating grid structures;

Task 2: Identify and implement a micromachining technique for manufacturing the negative collimator replicas (performed by NASA/JPL).

Task 3: Develop a CVD technique and processing parameters suitable for the complete tungsten densification of the collimator replicas.

Task 4: Develop a chemical etching technique for the removal of the collimator replicas after the tungsten deposition process.

Task 5: Fabricate and deliver tungsten collimating grid specimens.

EXPERIMENTAL APPROACH AND RESULTS

A preliminary search for information regarding substrate materials and fabrication techniques for the negative replica indicated that the micromachining of single-crystal silicon was the best choice, as technology from the semiconductor industry could be easily applied. Tungsten was chosen as the grid material due to its high Z number and the advanced state of its deposition technology.

Micromachining of slits in the single-crystal silicon substrate was performed at NASA/JPL. A small circular saw with diamond wafering blades was used to produce slits with a width of 45 microns and an aspect ratio of >20:1. Figure 1 shows a schematic of the micromachined negative replica.

For the deposition of tungsten inside the machined slits, via the hydrogen reduction of tungsten hexafluoride (WF_6), the silicon replica substrate was placed between the two halves of a graphite holder, sitting on the bottom half and with a 0.005" clearance below the top half, and positioned exactly one inch from each end of the holder. This setup, shown in Figure 2, would force the reactant gas flow through the slits of the replica. The replica and the holders were placed inside a 5" diameter quartz chamber, which was pumped down to 5 torr and leak checked (to prevent unwanted foreign elements from entering into the system), then resistively heated to 400-500°C in an argon atmosphere at 200 cc/min. When the substrate reached 400°C, the tungsten and hydrogen flows were introduced at rates of 50 and 400 cc/min respectively. The direction of the gas flows was alternated every five minutes in order to obtain uniformity in the deposit. Due to the extremely low deposition rate (0.72 μm/hr), the total deposition time was 55 hours, during which the pressure, substrate temperature, and gas flows were kept constant. To prevent bonding between the part and holders, the system was disassembled, cleaned, and reassembled after every six hours of run time. A schematic of the overall CVD apparatus for tungsten deposition is shown in Figure 3.

Stable metallic tungsten coatings were deposited inside the machined slits, as shown in Figure 4, using the hydrogen reduction of WF_6. This is the "standard" tungsten hexafluoride process, which has long been used to deposit tungsten by CVD. However, due to the size of the slits, this reaction had to be slowed down by lowering the deposition temperature by 30% and increasing the $W:H_2$ ratio from 1:4 to 1:8. These changes reduced the chemical reduction of the WF_6 by the silicon substrate (i.e. etching of the substrate by the WF_6), shown in Figure 5, and yielded an extraordinarily uniform coating distribution as shown in Figure 6.

Incomplete filling of the slits, shown in Figure 7, was due to the fact that the tungsten deposit formed by "growing" from the side walls of the slits. When the growth from each side wall met, the inner void was sealed off. However, this should have very little effect, if any, on the performance of the collimator grid, since the void is only 2-4 μm wide.

In addition to a silane chloride gas etching method developed by JPL, Ultramet developed a new and less complicated technique using a 70% potassium hydroxide/30% sodium hydroxide solution at a constant temperature of 40°C. The etching of the replica by the hydroxide method yielded a clean, silicon-free tungsten collimator grid structure as shown in Figure 4. This is now a well-established technique for future use.

CONCLUSIONS AND RECOMMENDATIONS

- Silicon and tungsten were chosen as the replica and collimator materials respectively, due mainly to the small difference between their thermal expansion coefficients.
- The micromachining method proved to be inadequate for producing "fine" collimators, but was very efficient for "coarse" collimators with a grid pitch of 0.155 mm or greater.
- Tungsten was successfully deposited within the slits by CVD.
- A hydroxide solution method for etching the silicon substrate was developed by Ultramet, equally or more efficient than the silane chloride gas method developed by JPL.
- A substrate material that will not react with the deposition gases is recommended in order to maintain better control over the final dimensional tolerances.
- From the literature search and the experience obtained in this project, X-ray lithography is recommended for manufacturing of fine collimators (e.g. 0.035-mm pitch).

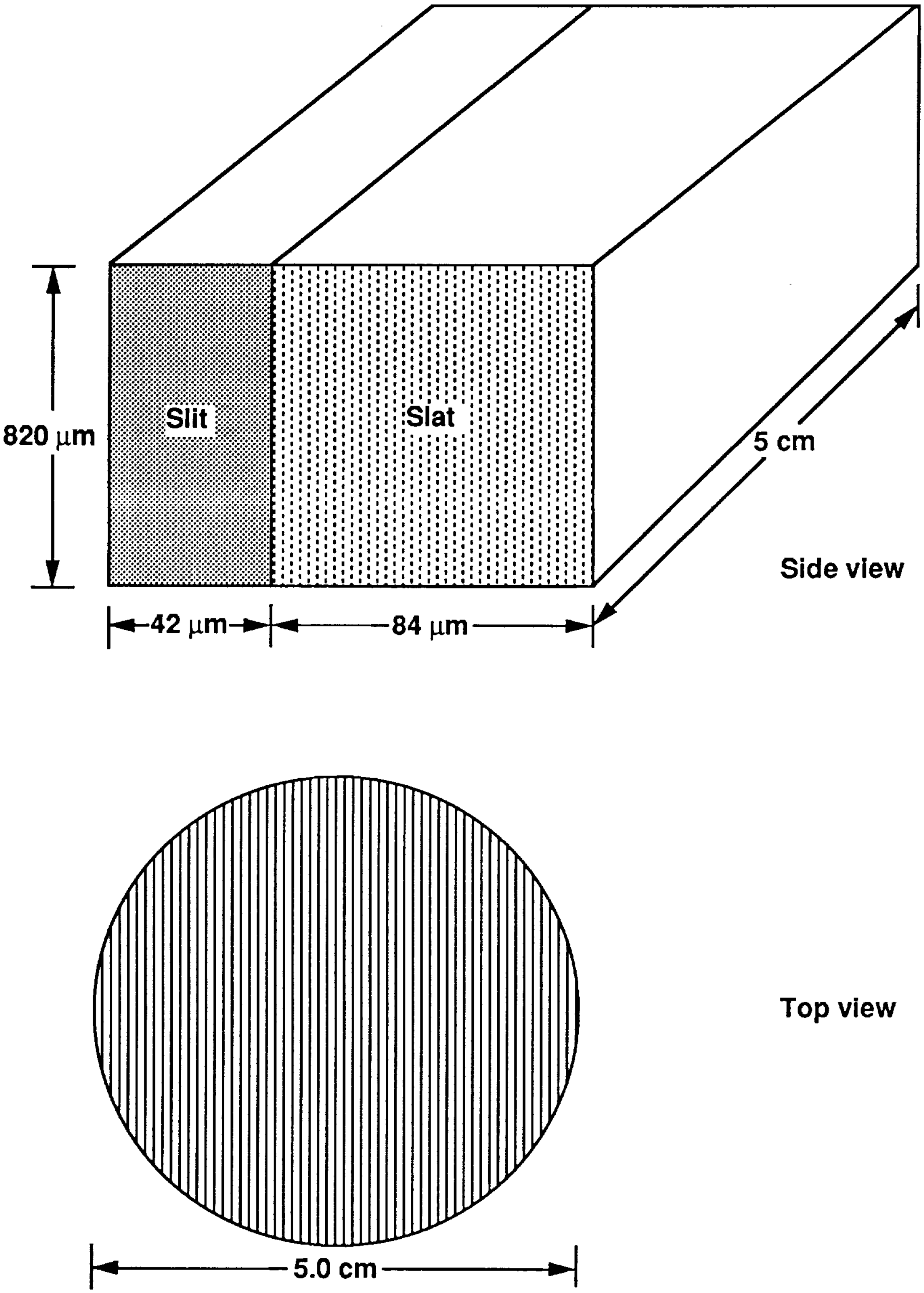
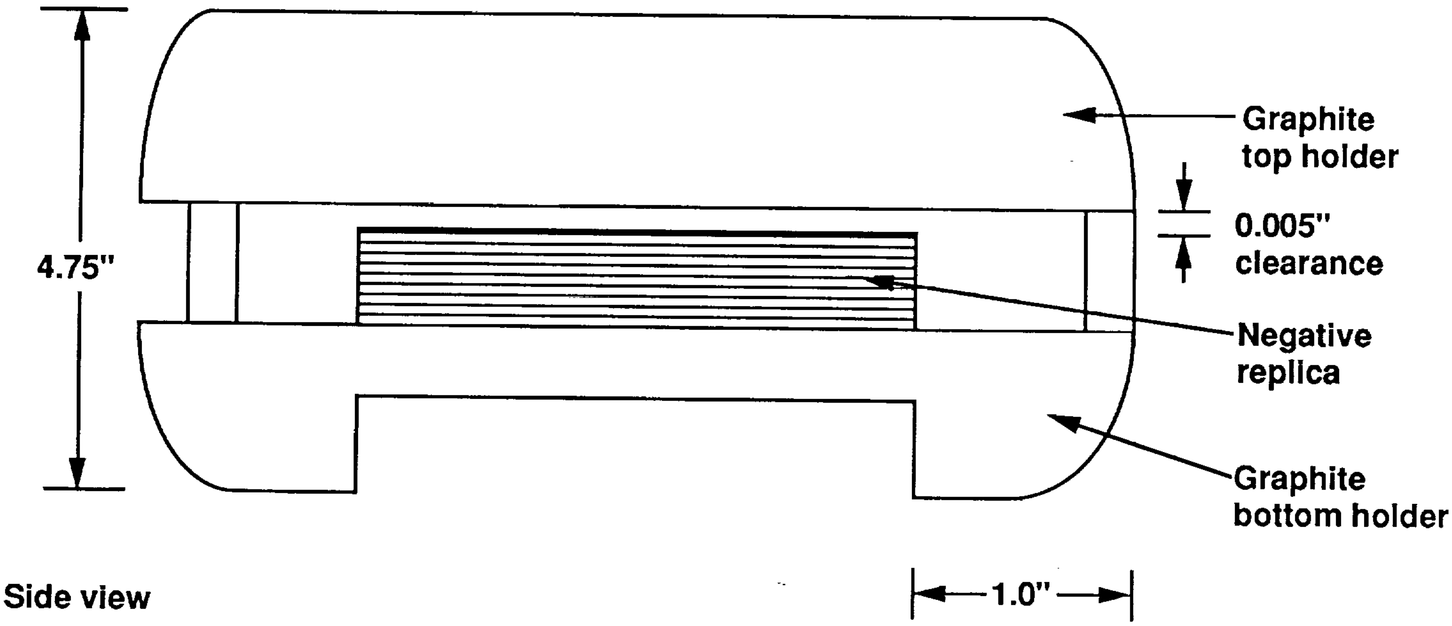


Figure 1. Schematic of micromachined silicon negative replica



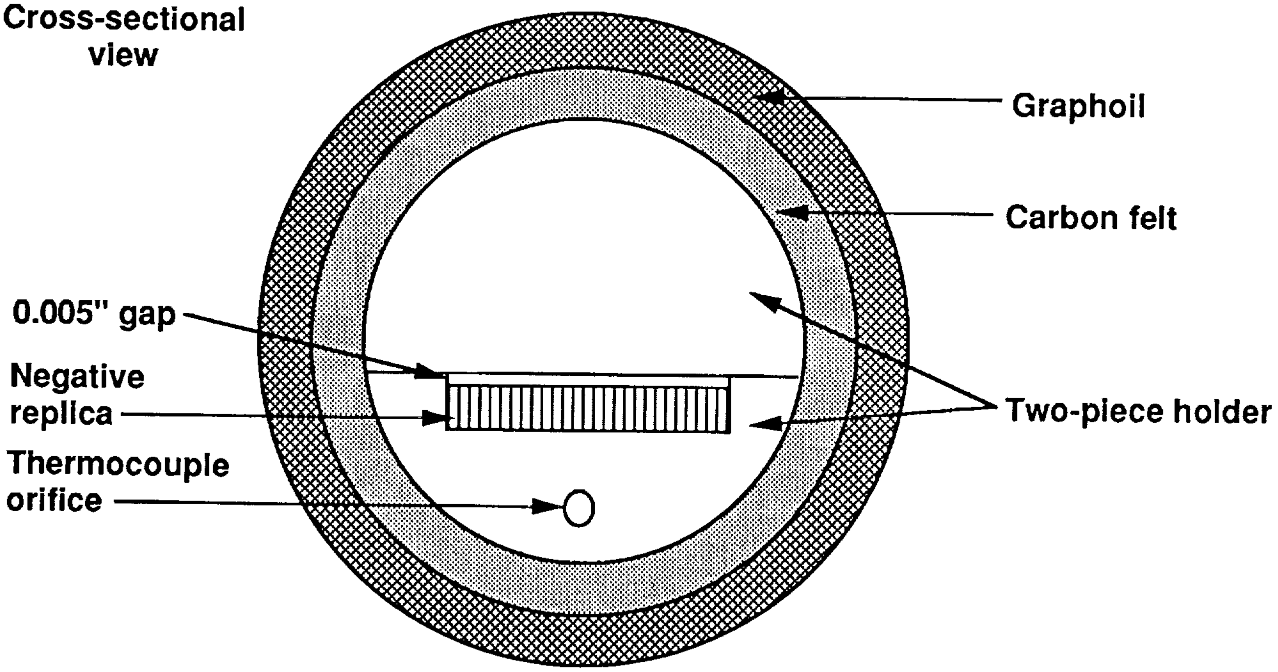


Figure 2. Schematic of negative replica/graphite holder setup

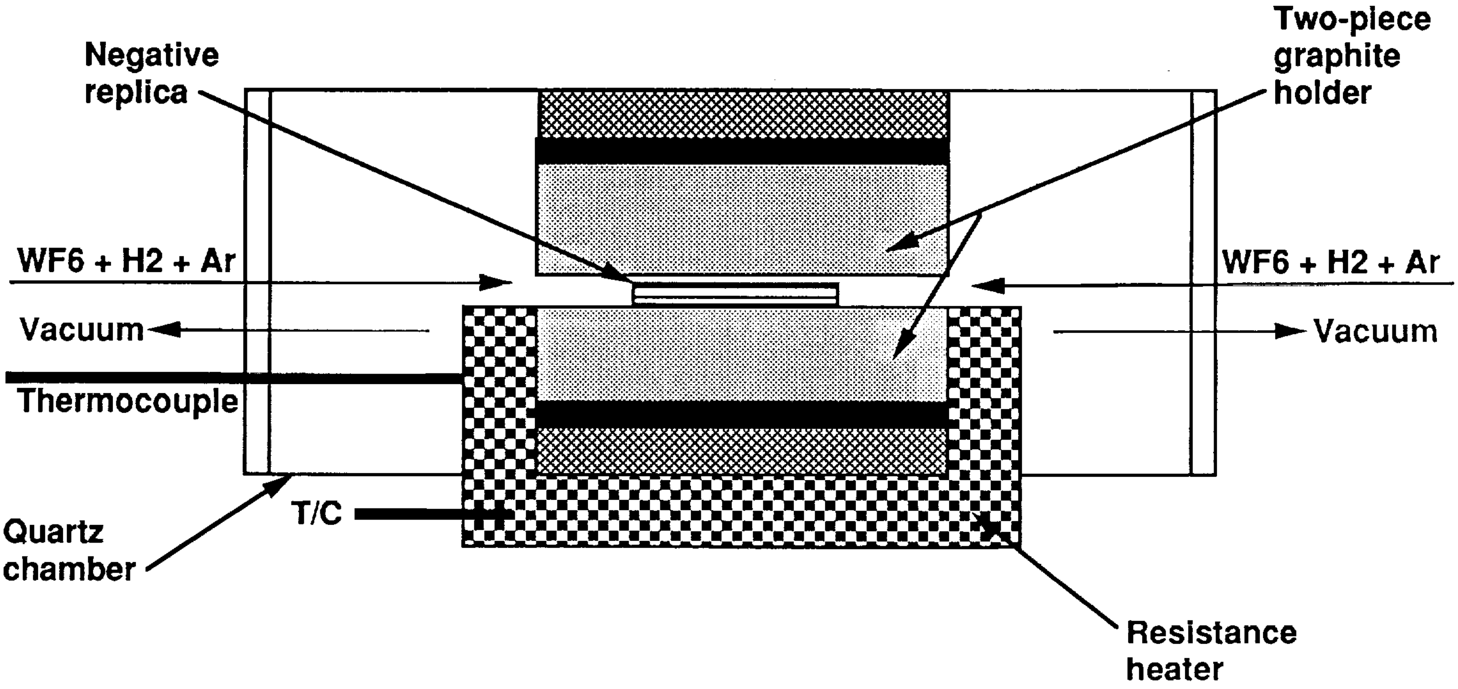


Figure 3. Schematic of CVD apparatus for tungsten deposition

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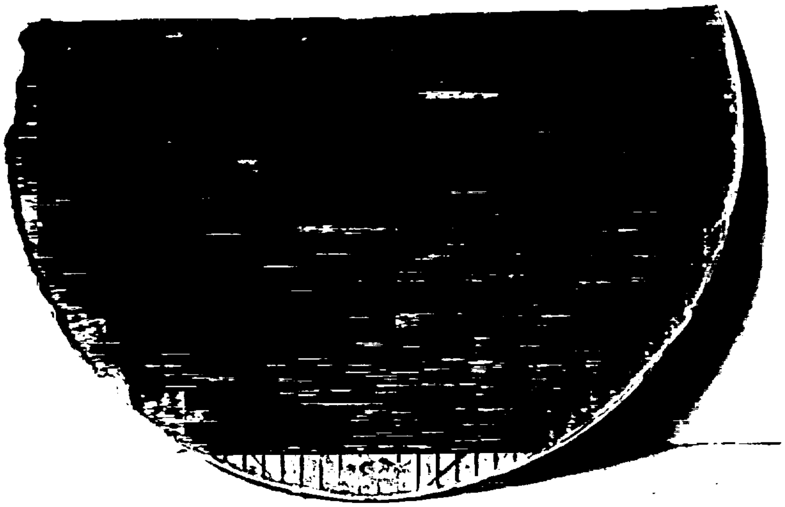

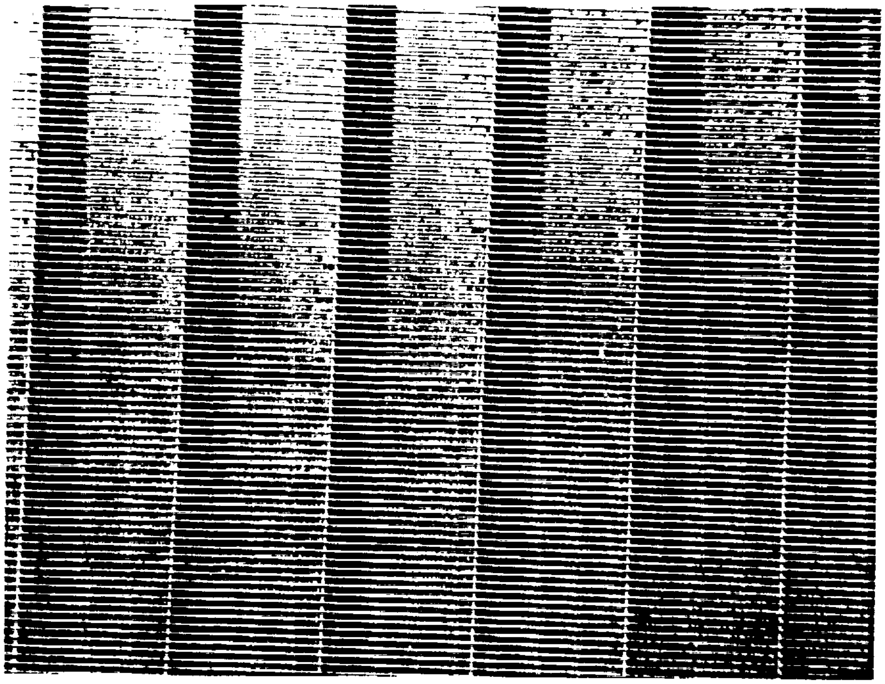

Figure 4. Final tungsten collimator grid structure (top: full part, actual size ≈2" diameter; bottom: optical micrograph, 10x)

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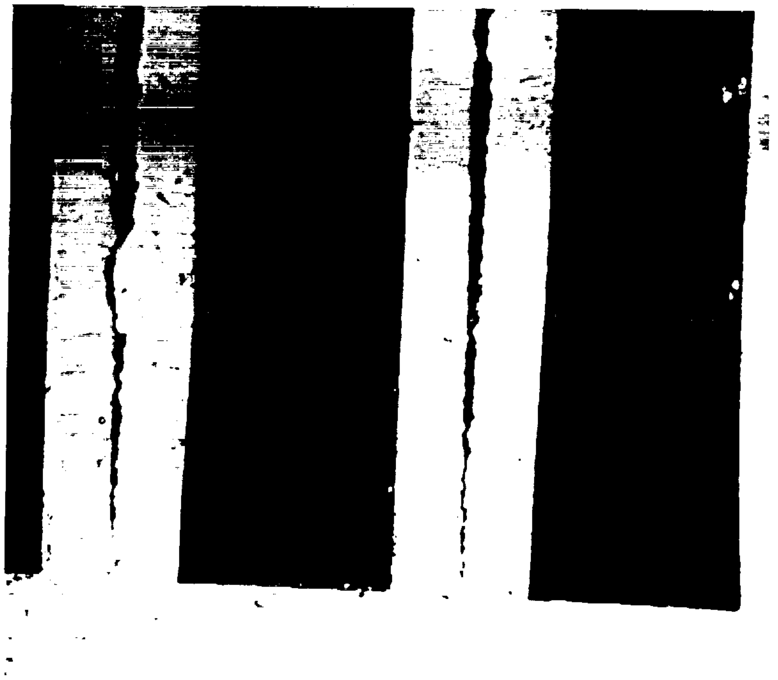

Figure 5. Optical micrograph showing etching of silicon substrate by WF_6

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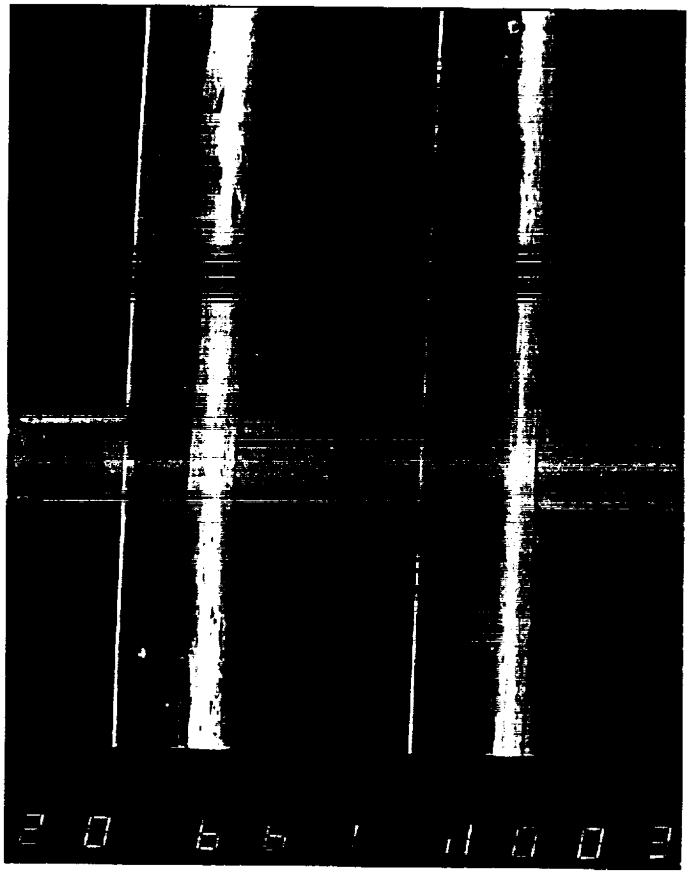

Figure 6. Scanning electron micrograph showing uniform tungsten coating distribution (660x)

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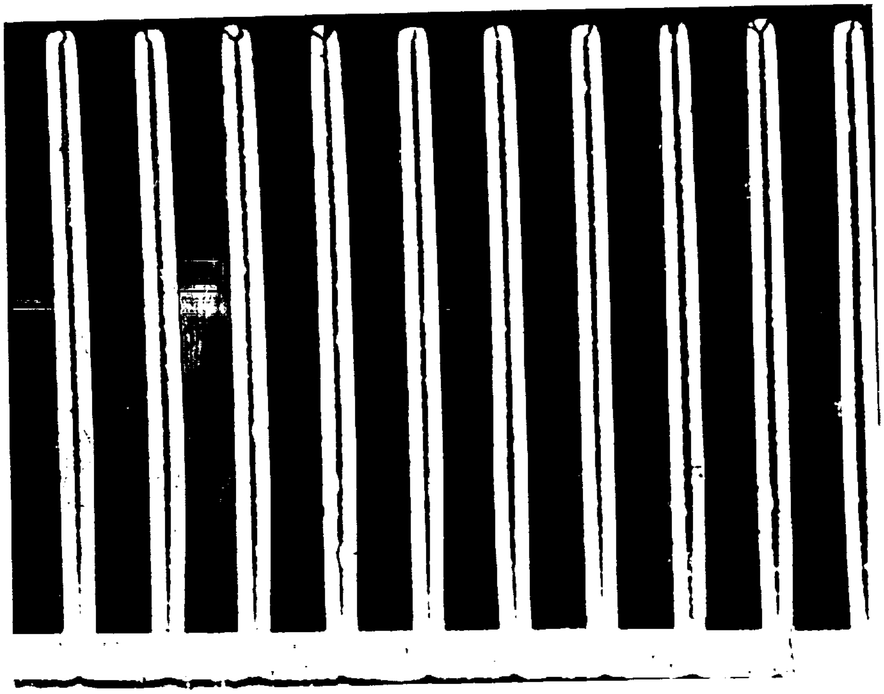

Figure 7. Optical micrograph showing incomplete filling of slits (voids within tungsten)

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Several upcoming missions will deploy X-ray and gamma-ray imaging systems that image specific wavelengths of high-energy EM radiation. Accurate imaging systems require collimating grids that eliminate off-axis radiation and provide imaging of an extremely small arc angle. Fabrication techniques investigated previously have not been able to meet the demanding tolerances (typically 0.0001" or less) required to meet grid performance goals. The objective of this project was to demonstrate the fabrication of extremely tight tolerance collimating grids using a high-Z material, specifically tungsten. The approach taken was to fabricate grids by a replication method involving the coating of a silicon grid substrate with tungsten by chemical vapor deposition (CVD). A negative of the desired grid structure was fabricated in silicon using wafering techniques developed for the semiconductor industry and able to produce the required tolerances. A network of accurately spaced slots was machined into a single-crystal silicon surface. These slots were then filled with tungsten by CVD, via the hydrogen reduction of tungsten hexafluoride. Following tungsten deposition, the silicon negative was etched away to leave the tungsten collimating grid structure.

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