SRI International

APPLICATION OF MICROFABRICATION TECHNOLOGY TO THERMIONIC ENERGY CONVERSION

Progress Report 2

Covering the Period 1 November 1979 to 31 January 1980

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ABSTRACT

Two diode designs are being studied that require basically different fabrication techniques.

The fabrication of electrodes was begun to test the concepts and the feasibility of these designs. This work was done with polished molybdenum electrode surfaces and vapor-deposited alumina insulator structures. Initial results were obtained in the close spacing of molybdenum electrodes with alumina insulators. Spacings were demonstrated with 1/2-inch diameter discs in the range of 3 to 5 μm , using 1- μm thick insulators. We have also demonstrated the deposition of thin alumina films on a molybdenum substrate, upon which arrays of thin film electrodes about 1/32-inch in diameter were deposited.

A second approach uses a thin film as one of the electrodes. An analysis of the thermal and electrical transport properties of 1- μm thick tungsten film shows that edge-connected films about 1 mm square in area could withstand the stresses generated by differential thermal expansion when the film is attached to the opposite electrode by stand-off insulators.

I INTRODUCTION

This is the second progress report in a theoretical and experimental program to evaluate those areas where three-dimensional microfabrication techniques could be important to improving methods of thermionic energy conversion. SRI International (SRI) is conducting the program for the Division of Fossil Fuel Utilization, Department of Energy (DOE) under Contract No. DE-AC02-79ET15423.

The scope of work encompasses a three year level-of-effort program to evaluate the significant of application of ultramicrofabrication techniques to thermionic energy conversion. To this end, the following specific steps will be attempted.

- Step 1: Prepare structure electrodes
- Step 2: Build and evaluate a submicron close-spaced diode
- Step 3: Study field-ion injection for space-charge neutralization
- Step 4: Conduct microfabrication service for programs in the thermionic energy conversion field.

The first year's effort is directed mainly toward Step 1, with some effort applied to Step 2. This report covers the period 1 November 1979 through 31 January 1980.

II PROGRESS DURING THE REPORTING PERIOD

The effort in the period covered by this report has been directed toward evaluating concepts for diode structures. Two diode designs with basically different fabrication techniques are being studied. Electrode fabrication was also begun to test the concepts and the feasibility of these designs. This work was done with polished molybdenum electrode surfaces and vapor-deposited alumina insulator structures.

Two of the principal problems that must be solved in a microstructure diode design are the maintenance of close spacing over large areas and the relief of stresses induced by the differential thermal expansion in the diode electrodes. One approach is to use a thin film as one electrode. An analysis of the thermal and electrical transport properties of 1- μ m thick tungsten film has shown that edge-connected films about 1 mm square in one area could withstand the stresses generated by differential thermal expansion when the film is attached to the opposite electrode by stand-off insulators.

Initial results were obtained in the close spacing of molybdenum electrodes with alumina insulators. Spacings were demonstrated with 1/2-inch diameter discs in the range of 3 to 5 μm , using 1 μm thick insulators. We also demonstrated the deposition of thin alumina films on a molybdenum substrate, upon which arrays of thin film electrodes about 1/32-inch in diameter were deposited.

III DISCUSSION

A. Diode Structures

1. Problems of Close Spacing

The placement of diode electrodes within close proximity requires a high degree of surface smoothness and surface flatness. When a 1- μm spacing is desired. The surface smoothness of the two electrodes must be a small fraction of a micrometer (rms). The flatness requirement is also stringent: For example, 1-cm diameter electrodes must have a surface radius of curvature much greater than 10 meters to maintain adequate spacing. Thus, a mirror-like polish with near-optical flatness is required.

Another major consideration is differential thermal expansion and its effects. If the insulator used to separate the electrodes is firmly anchored to both electrode surfaces, great shear stresses may be induced in the insulator as a result of differential thermal expansion of the electrodes. For example, the difference in linear expansion between a tungsten emitter at 1650°K and tungsten collector at 700°K is about 0.5 percent. Thus a 1-cm diameter diode would produce a differential expansion length of 50 μm . Clearly, some provision for relative motion or flexibility must be included in the diode design.

Some design concepts to account for problems of surface irregularities and differential thermal expansion are described in the next section.

2. Design Concepts

A straightforward approach to spacing the electrodes that skirts the problem of insulator strain is shown in Figure 1. The spacing is provided by a single insulator (or a closely spaced array) located at the center of the diode and anchored to only one of the electrodes. The assembly is held together by axial force applied at the center of the diode. Additional short insulators that do not span the gap may also be included at various radii to prevent the electrodes from touching in the case of slight warping during heating and cooling. This design requires a high degree of smoothness and flatness of the electrode surfaces. Machine polishing of metal surfaces often produces a slight convex curvature of the surface; therefore, in this design, the interelectrode gap might increase gradually with increasing distance from the center of the diode. Our initial results in fabricating this type of diode are described later in the report.

FIGURE 1 CONCEPT OF CENTER INSULATOR

FIGURE 2 CONCEPT OF DEPOSITED/ETCHED INSULATOR

A second approach that does not require a high degree of surface flatness is illustrated in Figure 2. An insulating film about $1\text{ }\mu\text{m}$ thick is vapor-deposited onto a solid polished electrode. The other electrode--also very thin--is a vapor-deposited metallic film. Lithographic techniques are then used to produce an array of small holes in the metal film and most of the insulating material is chemically etched away through these holes leaving an array of insulating posts in the gap. Finally, a heavier electrode for thermal and electrical connection is attached to the thin film electrode.

One possible representation of the final arrangement is shown in Figure 3. The advantage of this type of arrangement is that the closely spaced holes in the metallic film provide means to accommodate the differential thermal expansion by slight deformation of the film between the posts. The following analysis shows that the posts must be on approximate $10\text{-}\mu\text{m}$ centers to avoid film warping that might cause contact with the other electrodes. In addition, the electrical and thermal connections must be on a 1-mm grid to avoid large thermal gradients and IR voltage drop in the thin film. Thus, Figure 3 depicts only a section of a much larger array.

FIGURE 3 PORTION OF AN ARRAY USING THE CONCEPT SHOWN IN FIGURE 2

B. Electromechanical Analysis of Microstructured Diodes

A thin film used as the collector or the emitter in a diode must not only have sufficient strength to withstand the thermal expansion stresses, but must also be capable of transporting thermal power and electrical current from the free-standing film areas to the thermal and electrical connections. In this section we analyze these aspects to determine the spacing required for the support insulators and the thermal and electrical connections to the film.

1. Strain and Deformation

We assume here that the film electrode has a relatively large area and is supported on a large number of closely spaced insulator posts permanently attached to the other electrodes. When the film is the collector electrode, it will be stretched during operation; if the film is the emitter electrode, it will tend to be under compression and buckle during operation at normal temperatures. In the stretched case, the yield point of the film must not be reached. The tensile stress P is given by $P = Y(\Delta L/L_0)$, where Y is the elastic modulus and $\Delta L/L_0$ is the tensile strain. As noted previously, tensile strain for tungsten due to differential thermal expansion is about 0.5 percent.^{1,2} For tungsten, $Y = 5 \times 10^7$ lb/in². Thus the tensile stress, $P = 2.5 \times 10^5$ lb/in². This value may exceed the tensile strength of the film at 700°K.

2. Linear Film Expansion

Here we assume that the thin film is the emitter electrode. Thus, when the diode is heated, differential expansion causes the film to buckle because the emitter is hotter than the collector. We would like to know how far apart the insulating posts can be and still accommodate a buckling resulting from a 0.5-percent differential expansion. With a simplifying assumption that the film bends in the middle and deforms toward the opposite electrode, we find that (to avoid shorting) the distance between the insulating supports must be less than 20 times the interelectrode spacing. Thus, for a 1- μ m interelectrode spacing, the insulating posts must be on approximately 10- μ m centers.

3. Thermal Conduction

A circular diode that has heat supplied (or removed) at its periphery and removed (or supplied) uniformly over its surface area will have a temperature gradient across the surface caused by thermal conduction.

1. Y.S. Touloukian, R.K. Kirby, R.E. Taylor, and P.D. Desai, Thermal Expansion: Metallic Elements and Arrays (Thermophysical Properties of Matter, Vol. 12), Y.S. Touloukian and C.S. Ho. eds.
2. W.H. Kohl, "Handbook of Materials and Techniques for Vacuum Devices", (Rheinhold Publishing Corp. New York, 1967).

For a uniform heat flow through the surface, J_0 , and a thermal conduction coefficient, λ , the temperature differential between the center and edge of the film is given by $\Delta T = J_0 L_0^2 / 16 \lambda d$, where L_0 is the film diameter and d is the film thickness. This result shows that the thermal power cannot be transported over long distances through the film. For example, for $J_0 = 50 \text{ W/cm}^2$, $L_0 = 1 \text{ mm}$, $\lambda = 1 \text{ W/cm}^\circ\text{K}$, and $d = 1 \text{ }\mu\text{m}$, we obtain a temperature differential of about 300°K . Consequently, a device built using a film electrode must be supported on something like a honeycomb structure of the scale of a 1-mm grid to transport the heat adequately.

4. Electrical Conduction

It now remains to be determined whether the voltage drop resulting from electrical conduction through the film is significant. The voltage drop $\Delta V = J_0 \rho L_0^2 / 16 d$, where J_0 is the current density through the film surface, in A/cm^2 , and ρ is the electrical resistivity of the film material. Using the same diode example with $J_0 = 18 \text{ A/cm}^2$ and using $\rho = 16 \text{ }\mu\Omega\text{-cm}$ for tungsten, we arrive at $\Delta V = 1.8 \text{ mV}$. Thus, the voltage drop is much less significant than the temperature gradients in the film, because the temperature gradient will have a significant effect upon the distribution of emission over the emitter surface. For this reason, it would be more desirable to have the film as the collector electrode, where thermal gradients would have much less effect on the work function of the surface.

C. Electrode Fabrication Progress

For our initial studies of electrode fabrication, we purchased 1/2-inch diameter tungsten and molybdenum disks. The surfaces were ground flat and then polished with a polishing wheel. The disks were placed in a holder and rough polished with a Buehler nylon cloth and 6- μm diamond polishing compound lubricated with kerosene. Ten to twelve hours polishing time was required to remove the ground surface on the specimen.

Next a 1- μm diamond compound on a clean nylon cloth was used to remove any scratches made by the previous operation. This usually took two to three hours to clean up the surface. A final polish was then performed on a Buehler microcloth with Al_2O_3 polishing compound and distilled water for a clean scratch-free surface. The photograph of a polished molybdenum surface shown in Figure 4 was taken with interference contrast microscopy at a magnification of $162\times$. The dark sector in the right-hand portion of the picture is a 1- μm thick alumina film deposited upon the molybdenum surface. The interference contrast technique greatly emphasizes the apparent vertical structure of the surface. The vertical relief in this picture is less than about $0.1 \text{ }\mu\text{m}$.

An equivalent photograph for a polished tungsten surface is shown in Figure 5. For this case the vertical relief is somewhat greater than for the molybdenum sample; however, the most notable feature of our tungsten disks is the macroscale crack structure that covered the surface of

SCALE: 4mm ON PHOTO = 25 μ m ON OBJECT

FIGURE 4 MECHANICALLY POLISHED MOLYBDENUM SURFACE

SCALE: 4mm ON PHOTO = 25 μ m ON OBJECT

FIGURE 5 MECHANICALLY POLISHED TUNGSTEN SURFACE

most of these samples and that is evident in the figure. They are probably related to the technique used in manufacturing the disks.

To test the concept of separating two disks by small space, we deposited an array of aluminum oxide film on one of the disks as shown in Figure 6. We then placed another disk on top of this one, and determined that there was no shorting between the two disks. To determine the separation we measured the capacitance of the assembly. In one trial the average disk spacing was $3.2\text{ }\mu\text{m}$, and in a second trial it was $4.6\text{ }\mu\text{m}$. Even though the alumina film that divided the spacing was only $1\text{ }\mu\text{m}$ thick, we measured a much greater average disk spacing; this probably results from the disks not being perfectly flat, but rather slightly convex in their surface shape.

FIGURE 6 ELECTRODE PAIR, SHOWING PATTERN OF VAPOR-DEPOSITED ALUMINA SPACERS

In another fabrication test, a disk was completely coated with a $1\text{-}\mu\text{m}$ film of alumina, after which an array of $1/32$ -inch diameter aluminum disks was deposited as counterelectrodes. The purpose of this test was to determine whether the alumina film was pinhole free. (In films with pinholes, the counterelectrode deposition tends to short out the microcapacitor that has been formed.) The alumina disks were not shorted to the molybdenum. Capacitance measurements showed that the dielectric constant of the alumina film was within the expected range for alumina ceramic for one of the samples that we made; for two of the other samples, the measured dielectric constant was greater than that for alumina. These results may indicate that some tungsten oxide is being deposited from the vaporization source along with the alumina.

In summary, we have produced highly polished molybdenum and tungsten surfaces and deposited upon them $1\text{ }\mu\text{m}$ -thick films of alumina. We have also shown that counterelectrodes can be deposited on the alumina without shorting through to the opposite electrode. We have produced unshorted interelectrode spaces of 3 to $5\text{ }\mu\text{m}$, which can be reduced by producing a flatter electrode surface.

In the future, we plan to reproduce these results and to study further the characteristics of the alumina film.

IV PERCENTAGE OF TIME SPENT ON THIS EFFORT

During this reporting period, the Principal Investigator spent approximately 2 percent of his working time on this program. During the remainder of the contract, the Principal Investigator will devote approximately 400 hours of his time.