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MATERIALS PROBLEMS IN EVACUATED SOLAR ENERGY COLLECTORS

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Abstract.—An investigation of the absorptance and emittance of selective absorbing surfaces based on reactively sputtered metal carbide films is described. These surfaces exhibit good stability at 300°C in vacuum. Novel techniques have been developed for evaluation of absorptance and temperature dependent emittance of selective surfaces incorporated in assembled collectors. Thermal conduction losses in evacuated collectors due to low pressures of H₂, O₂, N₂, He, Ar, H₂ and CO have been determined. Methods for improving the efficiency of evacuated collectors, by using antireflection layers on the glass envelope or selective surfaces of enhanced absorptance are discussed.

1. Introduction.—Efficient heat extraction from non-concentrating solar collectors at temperatures 100-150°C may be achieved by vacuum insulation of the absorber element and by utilizing an absorbing surface of high selectivity. Evacuated tubular collectors are currently under development at Sydney University. In this paper, research connected with aspects of manufacture and testing of the collector is reviewed, with particular attention paid to the choice and treatment of materials used in the collector construction.

2. Collector design and selective surface production.—Prototype evacuated tubular solar energy collectors under development at Sydney University are manufactured from pyrex glass tubes of length ~ 1.4 m, absorber tube outer diameter either 22 mm or 30 mm and envelope outer diameter either 30 mm or 38 mm. The two tubes are assembled concentrically, (Fig.1) since this presents the minimum problem for mass production. The envelope is dimpled at three points to support the absorber tube in the concentric position. This design feature obviates the need for introducing foreign materials in the form of metal springs or clips to support the absorber tube.

The present production procedure involves baking the absorber tube in dry air at ~ 500°C prior to application of the selective surface, to reduce surface (and possibly bulk) contaminants of the glass. This procedure improves the prospects for rapid evacuation during the final assembly of the collector when the glass and selective surface are baked at ~ 500°C.
A selective surface developed at Sydney University specifically for all glass evacuated collectors consists of a reactively sputtered metal-carbide film deposited on sputtered copper. This selective surface was developed in small scale sputter coating systems 1, 2, 3/. A cylindrical magnetron sputtering system for coating batches of 20 tubes has now been operated successfully for 18 months, producing a total of 1,200 tubes 4, 5/. Figure 2 shows a schematic diagram of the sputter coater. The batch of twenty tubes is mounted on a carousel which produces planetary motion of the tubes about two magnetron sputtering sources. No change in the operating conditions of the coater are required when large (30 mm diameter) tubes are coated in place of the 22 mm diameter tubes, hence batches containing a mixture of both large and small diameter tubes have been coated successfully. Two magnetron sputtering electrodes, 38 mm diameter and 1.7 m long are suspended from the top plate of the chamber. One electrode consists of copper tube, the other

Fig. 2.- Schematic diagram of the magnetron sputter coating system. An axial magnetic field of ~ 0.02 T is applied during sputtering operations by a field coil wound on the vacuum chamber (not shown). A : copper electrode ; B : Stainless steel (or chromium) electrode ; C : Carousel ; D : Twenty glass tubes driven in planetary motion during sputtering operations ; E : Sample tube with plane glass slides attached ; F : Water cooled screen ; G : Reactive gas inlet pipe.
consists of type 316 stainless steel tube. A copper tube coated with ~ 1.5 mm of electroplated chromium has also been used in place of the stainless steel electrode. There is sufficient material stored in each of these electrodes to coat some 15,000 glass tubes. The cost of gases consumed during sputtering is negligible so the materials cost for the selective surface used is < 2 cents per tube. The electrodes are separated by a water cooled screen which prevents their cross contamination. The uniformity of the sputtered coating along the glass tubes is not perfect due to sputtering electrode end effects. The electrodes are somewhat longer than the glass tubes to reduce these effects. Figure 3 illustrates the deposition rate for copper along a glass tube.

![Deposition rate R vs. distance X along a glass tube for copper deposited at electrode current 20A and 600V. X = 0 corresponds to the lower end of the tube when in the sputtering chamber.](image)

Slightly lower rates are obtained at either end.

The stainless steel (or chromium) electrode is first sputtered for ~ 4 minutes at 18A and 650V in argon at pressure 0.5 Pa. This step cleans the electrode surface of metal-carbide compounds formed during the final stages of the selective surface deposition in the previous coating run, and produces a layer of reasonable adhesion on the glass tubes. The copper electrode is then sputtered in argon for 8 minutes at ~ 20A and 620V to produce a 320 nm thick film of low emittance copper on the tubes. The stainless steel (or chromium) electrode is then sputtered in an argon-acetylene mixture which results in deposition of a metal carbide film onto the copper coated tubes. A sputtering current of 17.5A at discharge voltage ~ 600V produces a film of thickness ~ 90nm on the twenty tubes in 6-7 minutes. Thus an average coating rate of 1 tube per minute for the complete surface is achieved in this production system, which is limited by the power supply presently available. Ho-
However, evaluation of the properties of the surfaces produced in this coating system should give a realistic guide to the surface properties obtained in production coating systems where coating rates of four or five tubes per minute are envisaged.

The present coating system is conservatively capable of one run per hour or ~ 500 tubes per day on a three shift basis. Larger coating systems which are effectively an extrapolation of the Sydney University system could be designed within a ~ 1.8 m diameter horizontally mounted chamber \( /6/ \). Figure 4 illustrates two possible designs incorporating one hundred and forty 30 mm diameter tubes in a single tube circle or two hundred and forty tubes in a double tube circle.

![Diagram of coating systems](image)

The latter system (Fig. 4b) may result in slightly higher efficiency of sputtered material utilization as well as a higher average coating rate and better uniformity of coating along the tubes. The latter system also allows incorporation of more than two electrodes. Three or four different electrode materials could be incorporated if a multi-layer coating were required. Either system run with total electrode current > 60A should allow a production capacity in excess of 5,000 tubes per day since the fraction of production time involved in pumpdown would be small.

3. Properties of the selective surfaces.— Specimens of selective surface are obtained in various coating runs by introducing a sample tube with eight plane glass slides attached at equal intervals along its 1.5 m length (Fig. 2). Two types of selective surface have been investigated.
Homogeneous chromium or stainless steel carbide surfaces are produced by maintaining a constant flow of acetylene into the sputtering chamber during the metal carbide deposition. The acetylene flow is adjusted to produce a metal carbide film of resistance $-10 \, \text{k}\Omega/\square$, and the sputtering time adjusted to produce a reflectance minimum at 0.90 $\mu\text{m}$ for a peak solar absorptance of $\alpha \approx 0.82$. Techniques for absorptance and room temperature emittance measurement are discussed elsewhere /1/. Emittance values of $\varepsilon \approx 2.3 \%$ at 300 K are obtained for the homogeneous surfaces. Figure 5 shows the reflectance "vs." wavelength for a typical homogeneous chromium carbide surface. Samples have been aged in a continuously pumped vacuum chamber at temperatures 200°C, 300°C and 400°C.

![Graph](image)

Fig. 5.- Reflectance (R) "vs." wavelength ($\lambda$) for a homogeneous chromium carbide on copper selective surface. Continuous line : after manufacture ($\alpha = 0.81, \varepsilon = 0.024$); Broken line : after 3,800 h at 400°C ($\alpha = 0.78, \varepsilon = 0.08$).

Figure 6 shows absorptance and emittance as a function of annealing time for typical homogeneous chromium carbide surfaces. The surfaces are relatively stable at temperatures up to 300°C. At 400°C the emittance degrades slowly due to interdiffusion at the copper-metal carbide interface. Almost identical behaviour has been observed for stainless steel carbide surfaces. Figure 5 shows the reflectance of a chromium carbide surface after 3,800 h at 400°C in vacuum. The optical thickness of the absorbing layer has increased probably due to diffusion of carbon into the copper at the interface.

Graded selective surfaces are produced by varying the acetylene flow into the sputtering zone during the metal carbide deposition. Absorptances considerably higher than the 80 % characteristic of homogeneous surfaces may be obtained. Figure 7 illustrates qualitatively two grading profiles for sputtered selective surfaces which have recently been investigated in a small magnetron sputtering system.
Fig. 6.- Absorptance (a) and room temperature emittance (ε) "vs." ageing time for homogeneous chromium carbide selective surfaces annealed in vacuum at 200°C, 300°C and 400°C. Continuous lines: absorptances; Broken lines: emittances.

Fig. 7.- Diagrams illustrating qualitatively two types of grading profile, MD grading and DMD grading. The density of dots illustrates the concentration of metal in the film and the graphs illustrate the variation of metal concentration (m) and distance (d) from the copper substrate.
These may be classified as MD and DMD surfaces. In MD surfaces the metal carbide film is graded from metal rich at the copper substrate to dielectric at the film surface by increasing the reactive gas flow from zero to a maximum during the deposition /2, 3/. DMD surfaces are graded from dielectric at the copper substrate to metal rich near the centre of the absorbing layer to dielectric at the film surface /7/. Absorptances \( a > 0.92 \) may be obtained for both grading profiles. Only MD graded surfaces have been studied in the tube coating system. Reactive gas may be admitted to the sputter zone of the batch coater according to any recipe by manually opening a fine needle valve. The flow rate of reactive gas into the chamber is measured by monitoring the pressure drop across a stainless steel capillary tube. A range of grading profiles has been investigated. For the surfaces of highest absorptance \( (a \sim 0.93-0.95) \) a range of emittances \( (\varepsilon \sim 0.030 \text{ to } \varepsilon \sim 0.045 \text{ at } 300\text{K}) \) may be obtained by varying the sputtering time for pure stainless steel (i.e. with no reactive gas) in the initial stage of the deposition /8/. The high emittance films produced in this way may be useful for limiting the stagnation temperatures of evacuated collectors, particularly when concentrators are used. Figure 8 shows reactive gas flow as a function of time for deposition of a stainless steel carbide surface of high absorptance \( (a \sim 0.94) \) and relatively low emittance \( (\varepsilon \sim 0.030) \).

![Flow rate of acetylene (f) into the sputtering chamber as a function of time (t) for deposition of two metal carbide selective surfaces. Solid line: \( a = 0.94, \varepsilon = 0.030 \); Broken line: \( a = 0.88, \varepsilon = 0.026 \).](image)

This grading profile is utilized in the majority of production runs for both stainless steel carbide and chromium carbide. The chromium carbide surfaces exhibit marginally lower absorptance /5, 9/. Figure 8 also shows the \( \text{C}_2\text{H}_2 \) flow used to produce a very low emittance surface (\( \varepsilon \sim \))
0.026 at 300K) with somewhat lower absorptance $\alpha \sim 0.88$. The reflectances for both these grading profiles are shown in figure 9.

![Graph showing reflectance vs. wavelength for two graded stainless steel carbide selective surfaces](image)

Fig. 9.- Reflectance ($R$) "vs." wavelength ($\lambda$) for two graded stainless steel carbide selective surface manufactured using the grading profiles illustrated in figure 8.

Small samples of selective surface have been aged in vacuum at 200°C, 300°C, 400°C and 500°C. Figure 10 shows absorptance and emittance "vs." ageing time for graded chromium surfaces. The surface is stable at 200°C and 300°C after an initial small decrease in absorptance.

![Graph showing absorptance and emittance vs. ageing time for graded chromium surfaces](image)

Fig. 10.- Absorptance ($\alpha$) and room temperature emittance ($\varepsilon$) "vs." ageing time for graded chromium carbide selective surfaces annealed in vacuum at 200°C, 300°C, 400°C and 500°C. Continuous lines : absorptances ; Broken lines : emittances.
Both absorptance and emittance deterioration occurs above 400°C. The absorptance decreases due to the graded film becoming more homogeneous as carbon diffuses within the absorbing layer. The absorptances tend towards $\alpha \approx 0.85$ which is more typical of a homogeneous surface. Stainless steel surfaces deteriorate slightly more rapidly than surfaces based on chromium carbide /5/.

4. Emittance measurements at elevated temperature.- Emittances at elevated temperature for selective surfaces deposited on glass tubes have been determined by placing the coated tube within a glass tubular vacuum chamber with water cooled walls, and inserting heater elements in the coated tube. Figure 11 shows a schematic representation of the apparatus.

![Fig. 11.- Apparatus for measurement of power radiated or thermally conducted between two concentric tubes. A : Open ended glass tube (coated or uncoated) ; B : Outer glass envelope ; C : Annular region, evacua-
ted or containing low pressure gas ; D : Water jacket ; E : Water inlet; F : Water outlet ; G : Central heater segment ; H : Outer heater seg-
ments ; I : Thermocouple and heater leads ; J : Electronic manometer ; K : Needle valve for gas inlet ; L : To diffusion pump.](image)

The power input to the heaters required to maintain the coated tube at static temperature allows evaluation of the absolute value of emittance /8/. Figure 12 shows the temperature dependence of emittance for homogeneous chromium and stainless steel carbide surfaces. The emittances are reversible for cycles between room temperature and 300°C. Heating the surfaces above 300°C results in an irreversible increase in emittance. Similarly, graded surfaces deteriorate at temperatures above 300°C. Figure 13 shows the emittance of a graded chromium carbide surface after manufacture and after cycling between 100°C and 400°C.
Fig. 12. - Emittance ($\varepsilon$) vs. temperature ($t$) for homogeneous metal carbide selective surfaces. The emittance of sputtered copper is included for comparison. Solid line: Sputtered copper; Long dashed line: data for chromium carbide obtained in first run to 400°C. Short dashed line: reversible curve obtained after several cycles between 100°C and 400°C; Continuous line with circles: data for stainless steel carbide obtained in first run to 400°C; Continuous line with triangles: data for stainless steel carbide obtained in third run to 400°C; Continuous line with squares: data obtained in fourth run to 400°C.

Repeated cycling results in the $\varepsilon$-T relationship tending toward a reversible curve. Similar results are obtained for stainless steel carbide surfaces /8/. The ageing data for small specimens and the $\varepsilon$-T data suggest that a diffusion mechanism, probably diffusion of carbon into the copper substrate, becomes important at temperatures above 300°C.

Emittances of the selective surface in fully assembled evacuated collectors (Fig. 1) have been determined by inserting heater elements within the absorber tube and water cooling the envelope /9/. Emittances have been determined immediately after assembly of the collector, prior to a 500°C bakeout, and immediately after the bakeout and final sealing of the envelope.
Fig. 13.- Emittance ($\varepsilon$) "vs." temperature ($T$) for a graded chromium carbide selective surface of relatively low emittance. The emittance of sputtered copper is included for comparison. Solid line: sputtered copper; solid line with circles: data obtained in first run to 400°C; Long dashed line: data obtained in second run to 400°C; Short dashed line: reversible curve obtained after several cycles between 100°C and 400°C.

Figure 14 shows emittance "vs." temperature before and after final bakeout for stainless steel carbide surfaces made with the grading profiles and reflectances illustrated in figures 8 and 9 respectively.

Fig. 14.- Emittance ($\varepsilon$) "vs." temperature ($T$) for graded stainless steel carbide surfaces coated onto tubes and assembled into evacuated modules. Solid line: very low emittance surface (see Figs. 8 and 9), before 500°C bakeout (lower curve) and after 500°C bakeout (upper curve); Broken line: Surface of high absorptance and relatively low emittance (see Figs. 8 and 9), before 500°C bakeout (lower curve) and after 500°C bakeout (upper curve).
The prolonged bakeout of the collectors at 500-520°C for ~ 1 1/2 hours during final evacuation results in a significant increase of emittance due to diffusion within the surface. However, after bakeout of the surface at 500°C, no further changes should occur during continuous operation of the collector at ~ 100°C, or extended stagnation at up to 300°C. This has been confirmed by measuring the emittance of a collector immediately after manufacture, and again after seven months stagnation outdoors at temperatures up to 280°C. In measuring the emittance after seven months stagnation, the collector envelope was fractured to allow removal of any gas which may have accumulated in the module.

5. Absorptance measurements for assembled collectors.- Efficiencies η₀ of evacuated collectors isolated by a highly absorbing background have been measured by comparing the temperature rises of probes inserted within the collector being investigated and within a calibrated "standard" collector while exposed to the same natural insolation. Construction of the temperature probe and the procedure for calibration of the standard collector are described elsewhere /9/. This strictly comparative measurement technique which obviates the need for a pyranometer and may be used successfully under steady state or intermittent insolation conditions, allows a sensitive evaluation of the absorptance of the selective surface at any time during the lifetime of the collector. A maximum efficiency η₀ = 0.835 has been measured for stainless steel carbide surfaces while η₀ = 0.815 for chromium carbide. The absorber area used in calculation of η₀ is equal to the cross sectional area of the absorber tube. The relationship between efficiency η₀ and absorptance α is discussed elsewhere /9/. By inserting the temperature probe at various points along an absorber tube, absorptance as a function of position has been evaluated. Figure 15 shows η₀ as a function of position along the absorber tube for a typical collector.

![Graph](image-url)
Slightly lower absorptances are measured at the extreme ends of the absorber tube due to end effects associated with the sputtering electrodes /8/. Efficiency measurements on a collector before and after bakeout at 500°C for ~ 1 ½ hours indicate a deterioration in absorptance of 1%. Efficiency measurements on a collector immediately after manufacture, then after seven months of stagnation outdoors at temperatures up to 280°C indicate an absorptance deterioration of less than 1%. 6. Collector degradation due to surface outgassing.- After prolonged operation at elevated temperature, gas may accumulate in the evacuated space of collector tubes. Possible processes causing gas accumulation are desorption from the glass and selective surface, decomposition of the selective surface and permeation of atmospheric helium through the glass absorber tube while at elevated temperature. The outgassing rate from baked pyrex is far superior to that for all other materials including stainless steel /10/ suggesting the advantage of all-glass evacuated collectors in comparison with metal absorbers in glass envelopes. Water is the predominant gas desorbed from the surface and bulk of the glass. Some of this is removed from the absorber tube prior to coating by a prebaking procedure in dry air. The glass envelope should also be baked prior to the final assembly of the collector. A preliminary analysis of gas pumped from the collector during bakeout at 500°C has indicated the presence of H₂O, CO, CO₂, O₂, N₂, and possibly argon and hydrogen. CO and CO₂ result from reaction of H₂O with the metal carbide coating. Small quantities of argon and hydrogen may be incorporated in metal carbide coatings during the sputter deposition. Getters have not yet been utilized in the Sydney University collector, however barium-aluminium based materials should getter all gases except argon and helium.

Thermal conduction losses between concentric glass tubes containing low pressure (0-1.5 Pa) Ar, CO, H₂, He, H₂O and N₂ have been measured using the apparatus shown in figure 11. In this pressure range for modules of the dimensions described, heat loss is proportional to pressure for all gases except H₂O. The theory for heat conduction through gases when the pressure is such that the mean free path is greater than the dimensions of the vessel predicts a linear dependence of heat loss on temperature difference and pressure /11/. Heat loss has been measured as a function of temperature for a plain glass inner tube, for a tube coated with sputtered copper, and for a tube with a complete graded stainless steel carbide selective surface. Conduction loss depends on the interaction of gas molecules with the surfaces, and hence
on the materials coated onto the glass tube. Figure 16 illustrates the trend observed for the three surfaces considered for helium gas. The same trend is observed qualitatively for all six gases. The selective surface results in maximum thermal conduction. This trend is partly due to the composition of the selective surface and may be partly due to increasing surface roughness of the sputtered film as thickness is increased.

Fig. 16.— Thermally conducted power (P) "vs." temperature difference (ΔT) between inner tube and water cooled glass envelope, for low pressures (0 - 1.5 Pa) of helium gas introduced between the tubes. Open circles : plain glass inner tube; Triangles : inner tube coated with 320 nm of sputtered copper; Closed circles : inner tube coated with graded stainless steel carbide selective surface.

If the atmospheric partial pressure of helium (~ 0.5 Pa) diffuses into the evacuated region, (as it will, if the module stagnates above 300°C for periods of the order of weeks /12/), a conduction loss of ~ 25 W per m² of absorber can be expected for an operating temperature of ΔT = 100°C. This conduction loss would reduce the heat extraction efficiency at 100°C with 1 kW/m² incident on the absorber area by about 3 %. Figure 17 illustrates conduction loss "vs." ΔT for all six gases for the selective surface coated onto the inner glass tube. These results suggest that the pressure of H₂O and H₂ in particular must be maintained below 0.5 Pa to maintain reasonable efficiency of collectors at operating temperatures above 100°C.

7. Methods of improving collector efficiency.— The absorptance of small samples of graded selective surface has been enhanced by ~ 2 % by depositing a light coating of carbon particles on the surface from a cool hydrocarbon flame /5/. Absorptances in excess of 96 % have been obtained, however the emittance is also increased by ~ 1 %. Carbon coatings are compatible with metal carbide surfaces, and although extremely fra-
gile, would be suitable for inclusion in evacuated collectors.

Fig. 17.- Thermally conducted power (P) "vs." temperature difference (ΔT) between inner tube and water cooled glass envelope for low pressure (0-1.5 Pa) of various gases introduced between the tubes. The inner tube is coated with a graded stainless steel carbide selective surface.

The efficiency of evacuated collectors could feasibly be enhanced by 3% to 5% by application of antireflection layers to the pyrex envelope. A promising antireflection layer consists of silica particles deposited from an alcoholic solution when the glass is withdrawn from the solution at speeds of 2-20 mm.s⁻¹/13, 14/. After dipping, the glass may be baked at 500°C to harden the film, a process compatible with the final bakeout of the Sydney University collector. Reflectances of 2.1% have been obtained for plane glass after treatment /13/. The reflectance rises to 3.1% after three months in air, possibly due to the film absorbing water. In evacuated tubes, the film coated on the inner surface of the envelope should be protected from such deterioration.

8. Alternative selective surfaces.- The production and properties of some alternative reactively sputtered selective surfaces deposited onto glass tubes are presently under investigation. A graded metal oxycarbide surface produced by sputtering a stainless steel electrode in argon-carbon monoxide has reasonable selective properties (Fig. 18), however this surface deteriorates during a 1 hour bakeout at 500°C in the final collector assembly stage.

Highly refractory metals such as titanium are probably necessary to obtain acceptable stability for metal oxy-carbides.

9. Conclusion.- Metal carbide surfaces deposited at high rate in a cylindrical magnetron sputtering system have excellent selective properties and good stability at 300°C in vacuum. These surfaces may be suitable for mass production of evacuated collectors. Investigation of thermal conduction losses in collectors containing low pressures of various gases has illustrated that conduction loss depends on the mate-
Fig. 18.- Reflectance \( R \) "vs." wavelength \( \lambda \) for a graded stainless steel oxy-carbide selective surface. Solid line : After manufacture (\( \alpha = 89 \% \) \( \varepsilon = 2.8 \% \) ) ; Broken line : After ~ 1 hour at 500°C (\( \alpha = 85 \% \), \( \varepsilon = 3.3 \% \) )

The enhancement of absorptance of selective surfaces by a deposit of particulate carbon may result in higher heat extraction efficiency for tubes running at low temperature or incorporated in concentrators.

Further research is aimed at the investigation of the high rate production and properties of alternative selective surfaces manufactured in the magnetron tube coating system. Study of the gases desorbed from selective surfaces during bakeout at high temperature and evaluation of collectors incorporating antireflection layers on the glass envelope is also in progress.

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