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**DEVELOPMENT OF HIGH EFFICIENCY
SOLAR COLLECTOR PLATES**

Progress Report, January 1, 1975—May 31, 1975

**T. Santala
R. Sabol**

June 16, 1975

Work Performed Under Contract No. E(11-1)-2600

**Texas Instruments, Incorporated
Attleboro, Massachusetts**

**ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy**

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SOLAR COLLECTOR PLATES**

By
Teuvo Santala
Ronald Sabol

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Progress Report
Covering period January 1, 1975 to May 31, 1975

June 16, 1975

Contract No. E(11-1)-2600

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ABSTRACT

Composite metal technology is used to manufacture intermetallic compound (IC) absorption surfaces and to combine them integrally with a composite metal tube-in-sheet collector plate. Heat treatment process for Al-Ni system was optimized and $\alpha \geq 0.95$ and $\epsilon \leq 0.3$ were obtained over a broad range of compounding conditions. Studies by SEM show that the Al-Ni IC surface is porous, spongelike and the dimensions of its fine structure are of the same magnitude as the wave length range of solar radiation, explaining at least partially the selectivity. Auger electron spectroscopy indicated that rather continuous aluminum oxide layer covers the fine structure of the Al-Ni.

Low carbon steel-copper composite has been shown to be a suitable candidate for the collector plate. Additional work is required to resolve the conflicting requirements of the formation of the IC surface and of the manufacturing of the composite metal tube-in-sheet collector plate.

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I SUMMARY

The objective of the project is to develop a composite metal solar collector plate in which an intermetallic absorption surface is integrally combined with a tube-in-sheet collector plate. The goal for the intermetallic absorption surface is solar absorptance $\alpha > 0.9$ and the infrared emittance $\epsilon < 0.3$. The goal for the tube-in-sheet collector plate development is to use the composite metals technology to combine materials and use them in quantities and locations which assure the best corrosion resistance and the most economic use of materials.

Certain intermetallic compounds in which aluminum is one of the component metals have a highly porous surface structure when they are formed by reacting the component metal layers thermally in solid state. Al-Fe and Al-Cr were shown to have a high solar absorptance $\alpha > 0.95$ but their infrared emittance is also high $\epsilon > 0.55$ resulting in low selectivity.

Aluminum-nickel intermetallic compounds exhibit high solar absorptance and also significant selectivity. To optimize the Al-Ni system the effects of the thickness of the aluminum layer and the temperature, atmosphere, and time of the compounding heat treatment were evaluated. Typical values, obtained under broad compounding conditions, were $\alpha \sim 0.95$ and $\epsilon \sim 0.3$. Under closely controlled process conditions $\alpha \sim 0.95$, $\epsilon \sim 0.20$ and $\alpha/\epsilon \sim 4.75$ can be achieved.

Scanning electron microscope (SEM) pictures at 5000x show that the dimensions of the fine structure of each compound is of the same magnitude as the wave length range of the incident solar radiation, and these surfaces absorb as black body cavities. The exact reason for the lower infrared emittance of the Al-Ni intermetallic compound has not yet been determined, but the fine structure of its surface is porous rather than dendritic as it is for the Al-Fe and Al-Cr surfaces. Consequently, it appears that the Al-Ni surface radiates in the infrared wave length region as a smoother surface than either Al-Fe or Al-Cr.

Samples of unimpregnated and impregnated Al-Fe and Al-Ni surfaces have been subjected to environmental testing for five months in a typical flat plate collector with one glass pane and for three months in an accelerated durability test in which the relative humidity at room temperature is cycled between 10-20% and 100% every eight hours. Visually observable changes were not detected in any of the Al-Ni intermetallic compound surfaces after either testing. The Al-Fe compound surfaces started to show macroscopic rust spots after the three month accelerated testing, but visually observable changes were not detected after the five months exposure in the flat plate collector enclosure.

Previous report indicated that of the five initially evaluated composite material systems only low carbon steel/copper/copper/low carbon steel could be inflated into a tube-in-sheet panel. Aluminum and nickel layers were incorporated into this material system for the formation of the intermetallic absorption surface, and material was bonded to manufacture 11" x 16" test collector plates. However, the incompatibility of the compounding and of the tube-in-sheet process heat treatment has caused blistering at the bond interface. As an addition necking and rupture of the thin Ni layer adjacent to the Al has occurred, resulting in a less desirable mixed Al-Fe and Al-Ni intermetallic compound surface on the test collector plate. The rupture of the Ni layer can be eliminated by proper modification of the bonding process parameters, and work is continuing to find a compromise heat treatment process which will satisfy the requirements of both the compounding and the tube-in-sheet process.

Test facilities have been completed to test up to sixteen separate 11" x 16" collector plates simultaneously.

II INTRODUCTION

A. OBJECTIVE

The objective of this program is: 1) to evaluate various intermetallic compounds, in which aluminum is one component metal and commercially available basemetals are used as the other component, for their suitability as absorber surfaces and; 2) to develop manufacturing technology to combine these surfaces integrally into a cost effective, durable, composite metal tube-in-sheet collector plate.

The goal for the intermetallic absorption surface is a) solar absorptance $\alpha > 0.9$ and b) the infrared emittance $\epsilon_{3-30\mu} < 0.3$.

B. RESEARCH PLAN AND METHODOLOGY

To achieve the objectives, the program is divided into three tasks:

Task 1. Development of intermetallic absorption surface.

The variables that are investigated to achieve the objectives for the intermetallic compound surfaces are: a) composition of the intermetallic compounds, b) compounding conditions and c) the effect of impregnation of the porous intermetallic surface with polymers and glasses.

Intermetallic compound surfaces are manufactured using composite metal technology. The solar absorptance is measured in a solar simulator by using a calorimetric technique and the infrared emittance is measured with Lion Research Corporation Model 25B Emissometer.

The impregnated and unimpregnated samples are subjected to environmental and accelerated cyclic humidity-temperature testing to establish the criteria for durability. This part of testing will be continued beyond the duration of ERDA Grant E(11-1)-2600.

Task 2. Development of collector material system and geometry.

Durable and economical material combinations are selected for the composite metal collector plate, and a manufacturing

process will be developed to combine the intermetallic absorption surface technology with the existing tube-in-sheet manufacturing technology.

As a part of this task a simple tube-in-sheet pattern will be developed and a tool will be built for a 1 sq. ft. tube-in-sheet collector plate, which allows the testing of the heat transfer characteristics and the durability of the collector plate material system.

A limited number of test collector plates will be manufactured and evaluated for performance and durability. This testing will continue beyond the duration of this grant to obtain significant information on corrosion.

Task 3. Tooling and manufacturing of pilot quantities of Solar collector plates.

Using information obtained in Tasks 1 and 2 optimum channel geometry will be designed and a tool will be built for manufacturing a limited number of 2 feet by 4 feet tube-in-sheet collector plates for evaluation at Texas Instruments and at other users as designated by ERDA.

Furthermore, Texas Instruments proposes to sell to prospective users pilot quantities of 2 feet by 4 feet solar collector panels for systems evaluation.

III PROGRESS REPORT

A. REVISED PROGRAM TASK SCHEDULE

Figure 1 REVISED PROGRAM TASK SCHEDULE

Original target date

 Revised target date

B. DEVELOPMENT OF ABSORPTION SURFACES

1. Development of the Intermetallic Compound Surfaces

a) Process development for Al-Ni intermetallic compounds

The semi-annual progress report NSF/RANN/SE/AER74-09036/PR/74/4 indicated that of the evaluated aluminum intermetallic compounds Al-Ni showed the most desirable characteristics, high solar absorptance and relatively low infrared emittance. Tables I and II repeat the results given in the semi-annual report. It was evident from those tables that: a) aluminum-nickel was the most promising, b) 0.0008" aluminum thickness before compounding gave generally better absorptance and emittance values, c) cracked city gas was the best atmosphere for compounding and d) that the lowest compounding temperature gave the best results. The information obtained in using two different aluminums, alloy 1100 and 4022 was inconclusive.

Additional tests were conducted to determine the effects of lower compounding temperature and the composition of the aluminum. Only the effect of the silicon additive in aluminum was of interest, because it was known to increase the temperature for the beginning of compounding. This could be desirable from the standpoint of process annealings. Table III summarizes the results of the last test series and Figure 2 combines in graphic form the results for 0.0008" thick aluminum from Tables I, II and III.

At lower temperatures and at shorter times the 4022 Al/270 Ni combinations start to show a noticeable decrease in the values of α and especially ϵ , indicating that the compounding has not been completed and visual inspection of samples compounded 15 min. at 1150°F do show uncompounded areas of aluminum. Consequently the measured values of α and ϵ represent the averages of the compounded and uncompounded areas.

The samples of 1100 Al/270 Ni compounded 15 min. at 1150°F have properties which are typical for all Al-Ni

Base Metal	4022 Aluminum Thickness	1/2 HOUR @ 1500°F IN THESE ATMOSPHERES					
		Air		Cracked City		Cracked Ammonia	
		α	ϵ	α	ϵ	α	ϵ
Low Carbon Steel	0.0004	0.99	0.55	0.95	0.56	0.98	0.55
	0.0008	0.99	0.65	0.94	0.58	0.95	0.55
	0.0012	0.93	0.53	0.90	0.67	0.91/0.93	0.60/0.58
430 Stainless	0.0004	0.98	0.51	0.82	0.58	0.44	0.61
	0.0008						
	0.0012	0.90	0.52	0.96	0.67		
201 Nickel	0.0004	0.64	0.32	0.87	0.26		
	0.0008	0.91	0.30	0.91	0.29		
	0.0012	0.94	0.30	0.90	0.33		
304 SS	0.0012	0.89	0.47	0.90	0.66		
	1100 Aluminum Thickness						
Low Carbon Steel	0.0008	0.93	0.54	0.93	0.72	0.90	0.66
	0.0008					0.97	0.56

TABLE I FIRST EVALUATION MATRIX TO SCREEN POTENTIAL MATERIAL COMBINATIONS FOR USE AS SOLAR COLLECTOR SURFACES

4022 Aluminum on 201 Nickel

Atmosphere	Aluminum Thickness	1350°F				1500°F				1650°F			
		15 min		60 min		7.5 min		30 min		5 min		20 min	
		α	ε	α	ε	α	ε	α	ε	α	ε	α	ε
Air	0.0008	0.93	0.32	0.86*	0.28*	0.85	0.32	0.86	0.35	0.79	0.36	0.79	0.35
	0.0012	0.93*	0.26*	0.96*	0.32*	0.96*	0.27*	0.78	0.32	0.80	0.32	0.74	0.39
Cracked City	0.0008	0.92*	0.31*	0.99*	0.27*	0.89*	0.27*	0.91*	0.30*	0.90	0.35	0.86	0.30
	0.0012	0.96*	0.32*	0.90	0.40	0.90*	0.30*	0.83	0.37	0.86*	0.27*	0.85	0.29
Cracked Ammonia	0.0008	0.94*	0.27*	0.92*	0.30*	0.87*	0.27*	0.85	0.29	0.84*	0.27*	0.86	0.31
	0.0012	0.89*	0.27*	0.86	0.33	0.82	0.31	0.81	0.31	0.78	0.33	0.76	0.32

TABLE II

EVALUATION MATRIX TO DETERMINE PROCESS
PARAMETERS FOR AL-Ni INTERMETALLIC COMPOUNDS
AN ASTERISK INDICATES THOSE SAMPLES WHICH WERE
PLACED IN ENVIRONMENTAL TESTING

Material Combination	Aluminum Thickness	Compounding Temperature and Time in a Cracked City Atmosphere					
		1150°F		1250°F		1350°F	
		15 min.	60 min.	15 min.	60 min.	15 min.	60 min.
4022 Aluminum/ 270 Nickel	0.0008"	0.81 0.13	0.90 0.33	0.97 0.21	0.91 0.38	0.94 0.29	0.95 0.28
1100 Aluminum/ 270 Nickel	0.0008"	0.95 0.29	0.95 0.30	0.93 0.30	0.97 0.30	0.97 0.30	0.99 0.33

TABLE III

EVALUATION MATRIX TO OPTIMIZE PROCESS
PARAMETERS FOR Al-Ni INTERMETALLIC
COMPOUNDS

Figure 2. Absorptance and emittance values of aluminum-nickel intermetallic compounds as function of temperature and time of compounding

4022 Al/270 or 201 Ni { O 15 - 7.5-5 min
 X 60 - 30 - 20 min
 1100 Al/270 Ni □ 15 and 60 min

samples indicating that the compounding process has proceeded to completion. The results confirm the effect of Si in Alloy 4022 as a retardant for the formation of the intermetallic compounds. At the higher temperatures the differences between the Al-Ni intermetallic compounds formed using 1100 and 4022 Al alloys are rather insignificant.

At the highest compounding temperatures, even at the shortened times, the absorptance values start to decline indicating that a different surface morphology and possibly different intermetallic compounds are formed.

It is evident that for both 1100 Al-270 Ni and 4022 Al-270 Ni pairs a rather broad temperature range exists at which high absorptance, $\alpha > 0.9$, and relatively low emittance, $\epsilon \sim 0.3$, can be obtained. This is desirable because it makes the material combinations more suited for large scale manufacturing.

The results obtained with 4022 Al-270 Ni at 1250°F for 15 min., where $\alpha = 0.97$, $\epsilon = 0.21$ and $\alpha/\epsilon = 4.6$ and at 1150°F for 15 min. where $\alpha = 0.81$, $\epsilon = 0.13$ and $\alpha/\epsilon = 6.2$, indicates that under very well defined process conditions it might be possible to obtain an improved version of Al-Ni intermetallic compound with substantially reduced infrared emittance.

To complete the initial evaluation matrix an aluminum-chromium laminate was produced by first electroplating 0.0008" chromium on 430 stainless steel substrate and then electron beam evaporating 0.0003" high purity aluminum onto the chromium. Samples were compounded at 1500°F for 30 minutes in air, cracked city gas and cracked ammonia. Solar absorptance and infrared emittance were measured and the average values of three specimens are shown in Table IV.

TABLE IV

Atmosphere	Solar absorptance α	Infrared emittance $\epsilon_{3-30\mu}$	α/ϵ
Air	0.90	0.62	1.45
Cracked City gas	0.97	0.60	1.62
Cracked Ammonia	0.94	0.48	1.96

Table IV Solar Absorptance and Infrared Emittance
of Aluminum-Chromium Intermetallic
Compounds, Compounded at 1500°F for
30 min.

Results indicate that although high absorptance can be achieved the infrared emittance is also high. In this respect Al-Cr intermetallic compound is similar to Al-Fe compounds. And because the manufacturing process is more cumbersome Al-Cr is the least desirable of all the evaluated intermetallic compounds for a large scale use. However, these surfaces could have good corrosion resistance, therefore, samples were introduced in the environmental and accelerated cyclic humidity test.

b) Surface morphology of the intermetallic compounds

Earlier investigation (1) had shown that the high solar absorptance of Fe_2Al_5 was due to the surface morphology. The Al-Ni intermetallic compounds were presumed to be selective by virtue of having a highly porous surface structure with pore dimensions of the same order as the wave length of solar radiation. These surfaces absorb as cavities, yet radiate in the infrared wave length region as flat surfaces (2).

Scanning Electron Microscope (SEM) pictures were taken of the various surfaces with 1000x and 5000x magnification,

Figures 3-15. All the surfaces have a highly porous structure, explaining the high solar absorptance. The basic difference between Al-Ni intermetallic compounds and the Al-Fe and Al-Cr intermetallic compounds is that the latter ones have a dendritic, crystalline structure, whereas the Al-Ni intermetallic compounds have a sponge-like structure in which the pores of the fine structure have dimensions equal to the wave length range of the solar radiation.

The Al-Ni intermetallic compounds formed at the lowest temperature, Figure 8, show an incomplete compounding, consequently the emittance value $\epsilon = 0.13$ is the weighted average of the intermetallic compound and metallic aluminum surfaces. The corresponding absorptance, $\alpha = 0.81$ is higher than would be expected, but the presence of an Al_2O_3 oxide layer might have an effect.

Surfaces of Al-Ni intermetallic formed at 1500°F and 1650°F show a coarsening fine structure which dimensionally exceeds the wave length range of the solar radiation, thus the slowly declining value of α . The surfaces also have a faceted, crystalline coarse structure, which evidently radiates in the infrared region as a flat surface. An attempt was made to study more closely the fine structure to determine its nature. To improve the resolution, surfaces were shaded by Au. However, the shading uniformly reduced the details across the surface, indicating that the intermetallic compound surfaces are covered with a rather continuous film of electron transparent material.

An Auger electron spectroscopy was used to determine qualitatively the composition of several intermetallic compound surfaces and all of them showed aluminum and oxygen as major elements at the surface. In none of the samples was the other component metal of the intermetallic compound, Ni, Fe or Cr, present. Due to the high temperatures and the types of the atmospheres used in compound formation an Al_2O_3 layer will always be present on the Al surface at the

a) 1000X

b) 5000X

Figure 3 Scanning Electron Microscope pictures of 4022 Al(0.0008")/LCS compounded at 1500°F for 30 minutes in cracked ammonia. $\alpha = 0.98$ $\epsilon = 0.55$

a) 1000X

b) 5000X

Figure 4 Scanning Electron Microscope pictures of 1100 Al (0.0008")/LCS compounded at 1500°F for 30 minutes in cracked city gas. $\alpha = 0.93$ $\epsilon = 0.72$

a) 1000X

b) 5000X

Figure 5 Scanning Electron Microscope pictures of 4022 Al (0.0012")/430 SS compounded at 1500°F for 30 minutes in cracked city gas. $\alpha = 0.96$ $\epsilon = 0.67$

a) 1000X

b) 5000X

Figure 6 Scanning Electron Microscope pictures
of 4022 Al (0.0012'')/304 SS compounded
at 1500°F for 30 minutes in air.
 $\alpha = 0.89$ $\epsilon = 0.47$

a) 1000X

b) 5000X

Figure 7 Scanning Electron Microscope pictures of evaporated 1100 Al(0.0003" onto electroplated chromium (0.0008") on 430 SS compounded at 1500°F for 30 minutes in cracked city gas.
 $\alpha = 0.99$ $\epsilon = 0.59$

a) 1000X

b) 5000X

Figure 8 Scanning Electron Microscope pictures of 4022 Al (0.0008")/270 Ni compounded at 1150°F for 15 minutes in cracked city gas. $\alpha = 0.81$ $\epsilon = 0.13$

a) 1000X

b) 5000X

Figure 9 Scanning Electron Microscope pictures of 4022 Al (0.0008")/270 Ni compounded at 1250°F for 15 minutes in cracked city gas. $\alpha = 0.97$ $\epsilon = 0.21$

a) 1000X

b) 5000X

Figure 10 Scanning Electron Microscope pictures of 4022 Al (0.0008")/270 Ni compounded at 1350°F for 60 minutes in cracked city gas. $\alpha = 0.95$ $\epsilon = 0.28$

a) 1000X

b) 5000X

Figure 11 Scanning Electron Microscope pictures of 1100 Al (0.0008")/270 Ni compounded at 1350°F for 60 minutes in cracked city gas. $\alpha = 0.99$ $\epsilon = 0.33$

a) 1000X

b) 5000X

Figure 12 Scanning Electron Microscope pictures of 4022 Al (0.0008")/201 Ni compounded at 1500°F for 30 minutes in cracked city gas. $\alpha = 0.91$ $\epsilon = 0.30$

a) 1000X

b) 5000X

Figure 13 Scanning Electron Microscope pictures of 4022 Al (0.0008")/201 Ni compounded at 1650°F for 20 minutes in cracked city gas. $\alpha = 0.86$ $\epsilon = 0.30$

a) 1000X

b) 5000X

Figure 14 Scanning Electron Microscope pictures
of 4022 Al (0.0004'')/201 Ni compounded
at 1500°F for 30 minutes in air.
 $\alpha = 0.64$ $\epsilon = 0.32$

a) 1000X

b) 5000X

Figure 15 Scanning Electron Microscope pictures
of 4022 Al (0.0012'')/201 Ni compounded
at 1500°F for 30 minutes in air.
 $\alpha = 0.94$ $\epsilon = 0.30$

beginning of the compound formation. This oxide layer is presumably ductile enough to form at least locally continuous film of oxide over the intermetallic compound surfaces. This was evident in the attempts made with Au shadowing for SEM.

The surface of 4022 Al/270 Ni, Figure 10 , compounded at 1350°F for 60 min. in cracked city gas atmosphere was sputtered in the Auger electron spectroscopy to remove the surface layer. The oxygen, aluminum and nickel were continuously scanned. Although the distance scale of the abscissa of Figure 16 is only very approximate, it shows that approximately 200Å thick layer of almost pure aluminum oxide covers the intermetallic compound. After the initial oxide layer, the relative values of the Al, O and Ni change only gradually because the surface is exceedingly uneven and, as an example, layers of aluminum oxide can penetrate deep into the structure.

The results of Auger electron spectroscopy cannot be readily used to determine the composition of the intermetallic compound below the oxide layer. For the determination of the composition, specimens and standards have been prepared for electron microprobe analysis.

c) Environmental and accelerated cyclic humidity testing of intermetallic compound surfaces.

Environmental and accelerated testing of the unimpregnated and impregnated intermetallic compound surface samples were continued. Additional specimens of unimpregnated Al-Cr and Al-Ni surfaces, and Al-Ni surfaces impregnated with 0.001" methyl metacrylate and with 0.002" RA3200 glass enamel were added into the testing. The RA3200 glass enamel slip was provided by Mr. Clifford Ruderer of Ferro Corporation.

Color slides of each specimen were made under standardized lighting conditions before introducing the samples into the test. A second set of slides will be made after retrieval

Figure 16. Depth profile of Auger electron spectroscopy peak intensities of oxygen, aluminum and nickel for a Al-Ni intermetallic compound surface.

of the samples from testing to obtain records of the change in visual appearance. This is done as an addition to the measurement of α and ϵ before and after testing.

First set of samples were retrieved from the accelerated cyclic humidity test 5/24/75, but the α and ϵ measurements have been delayed due to equipment transfer.

C. DEVELOPMENT OF COLLECTOR PLATE MATERIAL SYSTEM AND TUBE GEOMETRY

1. Development of Material System

The previous report NSF/RANN/SE/AER74-09036/PR/74/4 mentioned that of the five initially evaluated composite material systems for the tube-in-sheet collector plate only the low carbon steel/copper/copper/low carbon steel could be inflated.

For manufacturing of test plates an 11.5" wide, prebonded three layer material of 15% extra low phosphorus (XLP) copper/80% low carbon steel (LCS)/5% nickel was used. 4022 aluminum was bonded together with two layers of the above mentioned material to a 0.040" final thickness giving a combination of 0.001" Al/0.001" Ni/0.0165" LCS/0.0003" XLP Cu/0.0165" LCS/0.001" Ni. The adjacent Al/Ni pair is used for the inter-metallic compound formation, the nickel layer on the back side of the plate does not have a functional purpose but is the result of a simplified small lot bonding run. The 0.003" thick copper to copper pair will form the tube lining in the tube-in-sheet forming operation. The bonded material was trimmed to 11" width for the test plate inflation.

A tool was built for a tube pattern shown in Figure 17. The test collector plates will be used principally to evaluate the performance of the intermetallic compound absorption surface and the durability of the total collector plate material system. As an addition the collector plates will be used to provide design data for the 2' x 4' collector plates and, therefore, the fluid channel center to center dimensions were selected to be nearly the same as can be

Figure 17 11" x 16" Test Collector Plate
Tube-in-sheet Pattern

expected to be needed in the full size panel so that temperature gradients across the plate can be measured more easily. The corner to corner entry-exit configuration is used to simplify the problem of balanced fluid flow on a small pattern and it also offers advantages for manufacturing and installation. A series of test plates have been successfully pre-inflated but difficulties have been encountered due to the incompatibility of the process requirements for the tube-in-sheet manufacturing and the formation of the intermetallic compound.

Two basic problems have been encountered:

1. Necking and rupture of the Ni layer adjacent to the Al and
2. Blistering of the Cu/Cu interface.

The ruptured Ni layer before compound heat treatment is shown in Figure 18. The underlying reason, although not completely explained, is the difference in the work hardening characteristics of the Al and Ni and the process parameters (3). The reason for the cracking of the nickel is not caused by the properties of the Ni, because the Ni at the back surface, without the Al has the same process history as the Ni between the Al and LCS, but does not show any evidence of discontinuity.

This discontinuity of the Ni has an adverse effect, because during the compounding heat treatment both Al-Ni and Al-Fe intermetallic compounds are formed, Figure 19, and previous tests have shown that Al-Fe intermetallic compounds have substantially higher infrared emittance. Consequently, the emittance of the mixed Al-Ni Al-Fe surface would be expected to have an inferior emittance when compared to pure Al-Ni intermetallic compound surface.

The blistering at the Cu/Cu interface is caused by a gas evolution at the bond interface during the compounding heat treatment. The tube-in-sheet process has three basic steps:

Fig. 18 Photomicrograph (200X) of Al/Ni/LCS composite showing the rupture of the Ni layer.

Fig. 19 Photomicrograph (200X) of compounded Al/Ni/LCS composite showing alternate formation of Al-Ni and Al-Fe intermetallic compound.

- a) Pre-inflating of the tube pattern
- b) Heat treatment to form the intermetallic compound surface and to secure the Cu/Cu bond
- c) Final inflation of the tube pattern.

For step b) initially a temperature (1350°F) was selected which gave the optimum properties for the Al-Ni inter-metallic compound surface. As a result 1/2"-3/4" diameter blisters were formed at the Cu/Cu interface. Preliminary tests have indicated that by properly selecting the time-temperature-atmosphere profile of the heat treatment a good, sound bond interface could be obtained.

2. Testing facilities

Cooling fluid circulating system, inlet-outlet temperature measurement and recording system, and the collector frame for 16 test collectors and the fluid flow measurement system for 6 test collectors have been completed and are waiting for the test collector plates, which have been delayed due to technical difficulties described in previous chapters. Figure 20 shows the schematic layout and the significant technical data of the circulating system.

Water-ethylene glycol mixture will be used as a circulating fluid. Because the properties, especially the viscosity and density of the cooling fluid are influenced by the temperature, the flow from each flow-meter can be directed into a weighing container so that each instrument can be calibrated frequently.

The two constant temperature baths provide a supply of water at controlled temperatures to the collectors. The inlet temperature for all collectors will be kept uniform and constant. Exit water will be monitored for temperature and flow rate and then passed through the heat exchanger to dump excess heat before water is returned to the constant temperature baths.

Figure 20 Schematic lay-out of the circulating system for the 11" x 16" test collector plates.

D. TOOLING AND MATERIALS MANUFACTURING FOR PILOT QUANTITIES OF COLLECTOR PLATES.

No activity during the reporting period.

E. PROBLEMS ENCOUNTERED

Two basic areas of technical difficulty have been encountered in manufacturing of the test collector plates:

1. Necking and rupturing of the nickel layer below the aluminum and used for the formation of the Al-Ni intermetallic compound and
2. Blistering at the Cu/Cu interface during combined intermetallic compounding and tube-in-sheet process heat treatment.

The necking and the rupturing of the nickel was detected because the intermetallic compound surface had a striated appearance after the compounding heat treatment of the test collector plate. Metallographic cross section (Figure 18) confirmed that the Ni layer adjacent to the Al layer had necked and ruptured during the processing of the test collector plate material. The underlying reasons for the necking and rupturing, although not completely explained, are the difference in the work hardening characteristics of the Al and Ni and the used bonding process parameters⁽³⁾. It is obvious that the cracking of the nickel is not caused by the properties of the Ni alone, because the Ni at the back surface, without the Al has the same process history as the Ni between the Al and LCS, but does not show any evidence of discontinuity.

This discontinuity of the Ni has an adverse effect on the performance of the collector plate because during compounding heat treatment both Al-Ni and Al-Fe intermetallic compounds are formed (Figure 19) and previous tests have shown that compounds have substantially higher infrared emittance. Consequently, the emittance of the mixed intermetallic compound surface would be expected to have an

inferior emittance when compared to a pure Al-Ni compound surface. To alleviate this problem, new process parameters have to be determined and a new bonding run made to manufacture test collector plates for performance testing.

The blistering at the Cu/Cu interface is due to the incompatibility of the optimum process parameters for the formation of the intermetallic compound absorption surface and the manufacturing of the tube-in-sheet collector plate. The tube-in-sheet process has three basic steps:

- a) Pre-inflating of the tube pattern
- b) Heat treatment to form the intermetallic compound surface and to secure the Cu/Cu bond
- c) Final inflation of the tube pattern

For step b) initially a temperature (1350°F) was selected which has been determined to give the optimum properties for the Al-Ni intermetallic compound surface. As a result 1/2"-3/4" diameter blisters were formed at the Cu/Cu interface. The blistering is caused by a gas evolution at the bond interface during the compounding heat treatment. Preliminary tests have indicated that by properly selecting the time-temperature-atmosphere profile of the heat treatment, a good, sound bond interface could be obtained.

This problem, however, has caused a delay of 2-3 months for installation of test panels into durability testing. Substantial effort has to be exerted to determine the proper process parameters and additional bonding runs are necessary especially to eliminate the defect caused by the necking and rupturing of the nickel.

The existing material can and will be used to manufacture 11" x 16" test collector plates, so that durability data can be collected as soon as possible. Collector performance data, although it is not expected to be as good as for collectors with pure Al-Ni intermetallic compound absorption surface, will be collected to obtain reference values and to test the data collection procedure.

The measurement of solar absorptance of new samples and samples retrieved from the accelerated corrosion testing has been delayed due to transfer of the solar simulator to a new location. Management action has been initiated to speed up re-installation of the equipment.

F. PLANS FOR SECOND AND THIRD QUARTER OF 1975

1. Development of Absorption Surfaces

a) Intermetallic Compounds

- A study of the surface morphology of the Aluminum/Nickel intermetallic compound will be completed.
- A paper on "Selective Intermetallic Compound Surfaces" has been accepted and will be prepared and presented at the International Conference of the International Solar Energy Society, in Los Angeles, 7/28-8/1/75.

b) Durability Testing

- Testing of unimpregnated and impregnated intermetallic compound surface samples will be continued in the collector environment and in accelerated testing. Samples exposed 6 months will be retrieved, visual appearance will be recorded by color photography and α and ϵ will be measured.

2. Development of Collector plate Material System and Geometry

a) Manufacturing of Materials

- Major emphasis will be put on determining the conditions leading to the necking and rupture of the Ni layer adjacent to the Al. Especially the following items will be investigated:

- Thicknesses and the ratio of Al and Ni
- Type and process condition of the materials used
- Bonding and annealing parameters

The change in the program schedule caused by this additional investigation has been considered in the Revised Program Task Schedule (Figure 1).

- Additional bonding run will be completed to manufacture

material for the 11" x 16" test collector plates.

b) Manufacturing of Test Plates

- Major emphasis will be put on determining process conditions which will result in blister free tube-in-sheet collector plates and at the same time result in Al-Ni intermetallic compound surface with $\alpha > 0.9$ and $\epsilon \sim 0.3$.

Using the existing lot of material a suitable time-temperature-atmosphere reduction schedule will be established.

- 11" x 16" collector plates will be manufactured first from the existing material to start evaluating durability, to measure temperature gradients across the collector plate for final collector plate geometry, and to evaluate the testing and data recording system.
- Unimpregnated and impregnated 11" x 16" collector plates will be manufactured from the new material with correct structure and installed for performance and durability evaluation.

The changes caused by the additional work have been considered in the Revised Program Task Schedule (Figure 1).

3. Tooling and Material Manufacturing of Pilot Quantities of Collector Plates

- Tube geometry will be determined for the 2' x 4' collectors.
- Preparation of the expansion tool for the 2' x 4' collector plates will begin in the third quarter.
- Material combination, process parameters and scheduling of material manufacturing will be determined by the results of the investigation conducted to eliminate necking and rupture of the Ni layer.

G. ASSESSMENT OF PROSPECTS FOR FUTURE PROGRESS

All the necessary facilities for the durability and performance evaluation have been completed.

Although partially unexpected difficulties have been encountered in the manufacturing of the 11" x 16" test collector plate material and collector plates, we feel, based on our past experience, that problems of necking and rupturing of the

Ni layer and blistering of the Cu/Cu interface can be resolved. We know for certain, as an example, that by increasing the thickness of the Ni its necking and rupturing can be eliminated. The reduction of the Al layer has a similar effect. However, we want to select from all the available alternatives the one which is best suited for large scale manufacturing and is the most cost effective.

The limited information which has been collected in durability evaluation is insufficient to assess the long term performance. Due to the thermal stability of the intermetallic compound surfaces, we expect them to be competitive with other selective surfaces in evacuated collectors and other collectors where high surface temperatures are achieved. In a parallel effort we have shown that plates with an Al-Ni intermetallic compound surface can be welded into collector plates which are suitable for evacuated tubular collectors.

IV UTILIZATION

The concept of the intermetallic compound absorption surface, integral with a composite metal collector plate, has been developed under this grant. Low carbon steel is used as structural material and copper is used as the heat transfer fluid channel liner in the composite metal tube-in-sheet collector. This concept has attracted considerable attention, because it offers potentially good corrosion resistance in applications where, as an example, aluminum collector plates are not compatible with the rest of the circulatory system. Several inquiries have been received and over 50 of our semi-annual reports have been distributed outside of TI. However, the development work of the composite metal tube-in-sheet collector plate is in such an early state that we have elected to postpone the pursuit of any of the potential commercial applications until the development has progressed to more definite results.

In a parallel effort test collector plates with an Al-Ni intermetallic compound surface have been manufactured for the Corning baseline evacuated collector and a preliminary manufacturing process has been developed. However, the loss tests conducted at Corning have shown that the overall average infrared emittance of the collector plate has to be reduced to $\epsilon \leq 0.15$ to be competitive with some of the electroplated selective coatings. The intermetallic compound surfaces might have an advantage, however, because of their thermal stability and their insensitivity to the direction of the incident solar radiation.

General Electric has purchased four collectors with Al-Ni intermetallic compound surfaces for evaluation in focussing collectors.

Representatives of the Massachusetts Department of Commerce and Development have visited Texas Instruments to discuss TI's participation in a possible solar energy demonstration program in Massachusetts.

V CONCLUSIONS

The following conclusions can be drawn from the obtained results:

- Intermetallic compound surfaces formed in situ offer a viable alternative as absorber surfaces for solar collectors:
 - Higher absorptances $\alpha > 0.95$ can be achieved with Al-Fe, Al-Cr and Al-Ni intermetallic compounds.
 - Moderate selectivity, $\epsilon \sim 0.3$ and $\alpha/\epsilon \sim 3$ can be achieved under broad processing conditions with Al-Ni intermetallic compounds and $\epsilon \sim 0.2$ and $\alpha/\epsilon \sim 4.5$ can be obtained under closely controlled processing and compounding conditions.
- Low carbon steel/copper tube-in-sheet collector plate with Al-Ni intermetallic compound can be manufactured but conflicting process annealing requirements for the compound formation and for the tube-in-sheet processing will reduce the latitude in selection of process parameters.

VI REFERENCES

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VII APPENDIXES

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