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SOLAR ABSORPTION CHARACTERISTICS OF SEVERAL COATINGS AND SURFACE FINISHES

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The results of a study conduct several films potentially favorable fo are presented. Included in the invest electrodeposits, and anodized coating The results of this study show selective optical properties of any of nickel was its high susceptibility to d Electroplated black chrome generally varied considerably and was also rela had the greatest moisture resistance copper and steel substrates showed th any of the chemical conversion films	ed to determine the solar absorption characteristics of use as receiving surfaces in solar energy collectors gation were chemically produced black films, black s. ed that black nickel exhibited the best combination of he coatings studied. A serious drawback to black gradation in the presence of high moisture environments. exhibited high solar absorptivities, but the emissivity tively high under some conditions. The black chrome of any of the coatings tested. Black oxide coatings on e best combination of selective optical properties of studied.			
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PREFACE

This report records the results of the evaluation by Marshall Space Flight Center of various coatings and standard treatments of surfaces being designed as solar engery collectors. Although the investigation included both proprietary and non-proprietary electrodeposited coatings, this report does not constitute an endorsement of one product as opposed to another. It merely evaluates the properties of these coatings for the singular purpose of solar heating and cooling research.

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SOLAR ABSORPTION CHARACTERISTICS OF SEVERAL COATINGS AND SURFACE FINISHES

INTRODUCTION

From the standpoint of its abundance, availability, and non-polluting nature, the sun currently appears to be a most promising alternative source of energy. Although solar energy will inevitably be harnessed and converted to electrical energy for conventional and other specialized indirect uses, the most immediate and practical application, based on our present technology, appears to be that of heating and cooling buildings, and heating water. However, before solar energy can be utilized for any of these purposes, it must be collected to produce a heating effect and the heat transferred to a working fluid.

A device for collecting solar energy has several distinct components, one of which is a receiving surface which absorbs a large percentage of the sun's energy. Receiving surfaces are usually prepared by reducing the solar reflectivity of metals while maintaining their high infrared (IR) reflectivity (low emissivity). To produce such a surface change, either a black coating is applied by electrodeposition or the surface is blackened or colored by a chemical change. The surface treatment or coating is controlled to a thickness sufficient to make the metal surface optically black yet thin enough to retain its high IR reflectance. These optically selective surfaces are generally expressed in terms of an α_{s}/ϵ ratio, where α_{s} is solar absorptance and ϵ is thermal or IR emittance. The higher the ratio, the greater the efficiency of the surface in terms of collecting solar energy.

This report records the results of an investigation conducted to determine the optical characteristics of a number of standard surface treatments and coatings, including the effects of process variations designed to produce high α_s / ϵ ratios. The investigation included chemical surface treatments described in the literature and both proprietary and non-proprietary electrodeposited coatings. The effects of moisture on several of the coatings are reported along with their optical characteristics.

EXPERIMENTAL PROCEDURE

The three basic types of coatings included in the investigation were those produced by electroplating metals and metal alloys, chemical conversion of metal surfaces, and electrochemical conversion of the metal surface to their oxides (anodizing). The coatings were initially produced either by following directions of the manufacturer in case of proprietary processes or by directions given in the literature in case of non-proprietary processes. Eventually, procedures were varied on both proprietary and non-proprietary processes in an effort to produce surfaces with optimum optical characteristics.

All optical measurements were made with either a Gier Dunkle solar and IR reflectometer or a Beckman DK-2A Ratio Recording Spectrophotometer. The solar absorptance was determined by computing the difference between the solar reflectance and unity; the emittance was determined similarly by computing the difference between the IR reflectance and unity. The spectral reflectance measurements were produced with the Beckman Spectrophotometer. The moisture resistance of the coatings was established by subjecting coated test specimens to a high humidity environment (air at atmospheric pressure) controlled at a temperature of 310° K (98° F) to 311° K (100° F), and a relative humidity of 92 to 98 percent.

ELECTROPLATED COATINGS

BLACK NICKEL

Black nickel is an electrodeposited alloy composed of zinc and nickel with small amounts of carbon, nitrogen, and sulfur ⁽¹⁾. It has been used as a decorative coating for many years. The coating offers very little corrosion protection, and for this reason it is normally plated over corrosion resistant basis metals or over intermediate coatings such as bright nickel. More recently, black nickel has been included in the study of coatings which provide absorptive and radiative properties to regulate the temperature of space vehicles. Battelle Memorial Institute reported a solar absorptance value of 0.959 and a room temperature emittance of 0.686 for black nickel plated on aluminum in a study of the stability of this coating in a simulated space environment. ⁽²⁾ W. R. Wade and D. J. Progar reported similar optical results when black nickel was plated on aluminum at a current density of 1.0 to 2.0 milliamperes per square centimeter (mA/cm^{2*}) for a period of 20 minutes. ⁽³⁾ The reported optical characteristics of this coating also made it attractive for solar collector application, especially the high solar absorptivity. The relatively high emissivity, however, was obviously a serious drawback if a high efficiency coating (high α_s / ϵ) was to be produced. Reduction of this high emissivity without significantly lowering the solar absorptivity was, therefore, the primary objective in the study of this coating.

After conventional cleaning, sample specimens of either aluminum or steel were first bright nickel plated to a thickness of around 0.0127 millimeters (0.0005 inch) to furnish both corrosion protection and a low emissivity base ($\epsilon = 0.06$) upon which to electroplate the black nickel. The black nickel was then plated under varying experimental conditions in a bath containing the following chemicals:

Nickel Sulfate (NiSO₄ . $6H_2O$)---- 75 grams

per liter (10 ounces per gallon) Nickel Ammonium Sulfate (NiSO₄ (NH4)₂ SO₄ . 6H₂O) - - - 45 grams per liter (6 ounces per gallon) Zinc Sulfate (ZnSO₄ . 7H₂O) - - - - - - - 37.5 grams per liter (5 ounces per gallon) Sodium Thiocyanate (NaCNS) - - - - - 15 grams

per liter (2 ounces per gallon)

Bath parameters such as temperature, current density, time, and pH were varied in an effort to produce the desired optical characteristics. Varying the current density and time of plating produced the most dramatic change in the optical properties of the coating since these variables relate directly to film thickness and to composition in the case of alloy plating such as black nickel. Plating times of 30 to 60 seconds at current densities of $4mA/cm^2$ produced the optimum coating with regard to optical selectivity. These plating conditions produced a film 1500 to 3000 angstroms ($\stackrel{0}{A}$) thick (Figure 1). Time periods significantly beyond 60 seconds produced a film with a thickness that decreased the IR transparency (raised the emissivity) without gaining any significant increase in solar absorptivity as indicated in Figure 2. A bath temperature of $300^{\circ}K$ ($80^{\circ}F$) and a pH range of 5.6 to 5.9 appeared to be the optimum for these variables although slight variations produced no significant optical change in the film. The films produced under these conditions exhibited absorptance and emittance values ranging from 0. 88 to 0.94 and 0.05 to 0.07, respectively.

In a solar residential heating and cooling system development program conducted by NASA at the Marshall Space Flight Center, 211 aluminum heat exchanger panels, 2 ft. wide x 3 ft. long, were black nickel plated under the above conditions. ⁽⁴⁾ The average solar absorptance and thermal emittance values for the black nickel film on these panels were 0.92 and 0.06, respectively, with an α_s / ϵ ratio of 15.3. The spectral reflectance in the solar region of the spectrum of a representative sample of black nickel film is shown in Figure 3.

Results of moisture tests showed that black nickel has good resistance to an environment controlled at around 50 percent relative humidity $[300^{\circ}K(80^{\circ}F)]$, but has a distinct lack of resistance to the high humidity environment previously described. Test panels exposed to the 50 percent relative humidity environment showed no optical degradation after 12 months exposure. Those exposed to the high moisture environment degraded from an initial solar absorptance of 0.90 to 0.74 in 44 hours. The initial emittance of 0.05 remained unchanged. Even though the results of these tests showed that the black nickel film did not have good resistance to a high moisture environment, it was used in the MSFC program because of the desirable optical characteristics. However, the humidity inside the collector was controlled to a level at which no significant optical degradation occurred. This was achieved by incorporating an active dry air purge in the collector.

Tests were also conducted to determine the long term effects of ultraviolet irradiation on black nickel. The test was carried out by first determining the optical properties of a plated sample and then exposing it to water-filtered light produced from a xenon lamp with an intensity of 0.8 solar constant (simulated sunlight). The test panel was exposed in an open laboratory environment at ambient temperature. The optical properties were re-determined periodically and compared with the initial values. As shown in Table I, after an exposure time of 8640 equivalent sunshine hours (ESH), the α_s and ϵ changed from an initial 0.89 and 0.06 to 0.83 and 0.08 respectively. No significant change in appearance occurred during this test period.

BLACK CHROME

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Black chrome is also produced by electrodeposition. Generally the deposit consists of around 75 percent chromium and 25 percent chromium oxide, although some proprietary baths are reported to produce deposits with substantially smaller amounts of elemental chromium. Like black nickel, it has been used primarily as a decorative black coating. It has also been used in applications where non-glare, low reflective finishes are desired. It is often plated over an initial deposit of bright nickel which provides both a smooth, bright base upon which to plate the black chrome, and a multilayer system which furnishes better corrosion resistance. It can, however, be plated directly over other metal substrates such as copper, stainless steel and bright chromium (the latter two require special activation for best adhesion).

In addition to decorative applications, black chrome has recently been considered for use in solar collectors as a selective solar absorber coating. In this study, two commercial processes were evaluated for their optical characteristics and, to some extent, their resistance to moisture; one bath, "Econo-Chrome BK, "* was produced by Dupont Chemical Company and the other, "Chromonyx"**, by Harshaw Chemical Company. The plating was accomplished at a current density of 250 mA/cm^2 and a bath temperature of 297° K (75° F) for each of the processes. The concentration of the Dupont bath was 480 grams per liter (64 ounces per gallon) of the "Econo-Chrome BK" compound. ⁽⁵⁾ The Harshaw bath contained 27 percent by volume of the "Chromonyx" addition agent and 337.5 grams per liter (45 ounces per gallon) of chromic acid. ⁽⁶⁾ Both processes produced coatings with good selective solar absorption properties provided the plating time was controlled to keep the thickness of the coating below a certain level. Absorptance and emittance values of the Dupont coating (with varying plating times) ranged from 0.93 to 0.95 and 0.12 to 0.60, respectively. Absorptance and emittance values of these experiments are shown in Figure 4. The spectral reflectance of both the Harshaw and Dupont Chrome is shown in Figure 5.

Results of short term environmental tests indicate that both the Dupont and Harshaw Chrome have excellent resistance to moisture. Test panels of steel, bright nickel plated to an approximate thickness of 0.0127 millimeter (0.0005 inch) prior to black chrome plating, were subjected to a high moisture environment previously described. After three months exposure, no significant optical degradation nor change in appearance had occurred.

"ELECTROBLACK"***

"Electroblack" is a proprietary black electroplating process and may be applied to any metal that normally receives an electroplate. ⁽⁷⁾ The process is easy to control and is plated at a current density of from 5 to 60 mA/cm². The coating composition was not disclosed in the manufacturer's technical literature and no attempt was made in this study to determine its composition. Results of tests conducted to establish the selective solar absorption

^{*} Trade name of E. I. Dupont De Nemours and Company

^{**} Trade name of the Harshaw Chemical Company

^{***} Trade name of Enquest Chemical Co., Inc.

characteristics of "Electroblack' aré shown in Figures 6 and 7. Test specimens from which these optical values were derived were plated by the "Electroblack" process at a current density of 50 mA/cm² and a bath temperature of 343° K (157°F); the basis metal was brass. The coating was also applied to bright nickel over steel but the solar absorptivity in this case was generally lower than when applied to brass. From 30 to 45 seconds plating time appeared to produce the highest solar absorptivity and the lowest emissivity. Unlike black chrome and nickel, the solar absorptivity appeared to peak at about 45 seconds plating time and decreased with increased plating time. The emissivity increased with plating time as expected.

Test results showed the "Electroblack" has good resistance to moisture as evidenced by no degradation in solar absorptivity and only minor degradation (increase) in emissivity after exposure to a high humidity. After 4.5 months exposure, the solar absorptance increased from an initial 0.86, to 0.88 and the thermal emittance increased from an initial 0.08, to 0.13.

CHEMICAL CONVERSION COATINGS

"EBONOL C"*

This coating is produced by chemically converting the surface of copper and its alloys to cupric oxide (CuO) which is usually jet black in appearance and reportedly has good thermal and chemical stability. For decorative finishes and most other conventional applications, the blackening time is from 5 to 10 minutes in a bath containing 180 grams per liter (24 ounces per gallon) of the "Ebonol C" salts. ⁽⁸⁾ This produces a jet black finish but usually with the presence of a nap which can be removed by hand wiping with a cloth. A treatment time slightly less than one minute (or for a period of time that prevents the formation of nap) at this concentration appeared to

*Ebonol is a trade name of Enthone, Inc.

produce a coating with the most optically attractive properties with regard to absorbing solar energy. The solar absorptance and thermal emittance of this coating produced at different treatment times are shown in Figure 8. The spectral reflectance characteristics in the solar region are shown in Figure 9. As shown, increasing the treatment time gradually increases both the emissivity and absorptivity although the absorptance curve shows considerable fluctuation up to about one and one half minutes treating time. Treatment times beyond this point substantially increase the absorptivity, but they increase the emissivity to an undesirable level. The spectral reflectance curve (Figure 9) shows some oscillation in the 0.27 - 1.7 mm wavelength range (a significant portion of the solar region) which probably indicates a lack of absorption in this region resulting in an overall moderately low solar absorptance of around 0.84.

Results of moisture tests on this coating showed that it had moderately good resistance to a high humidity environment. This was evaluated on a test panel treated for 45 seconds in the above bath. There was no degradation in solar absorptivity and only slight degradation in emissivity after 120 days exposure. The solar absorptance increased from an initial 0.81, to 0.83 and the emittance increased from an initial 0.07, to 0.11.

"EBONOL S-34"

This coating is produced by chemically converting the surface of iron and steel to ferroferric oxide, Fe $(FeO_2)_2$, in a high temperature, highly concentrated alkaline bath. The process produces a shiny, uniform, jet black coating on buffed surfaces in about 3 to 25 minutes depending upon the steel alloy to be coated. For the purpose of this program, the operating temperature and bath concentration were maintained at the recommended range of 413° -422° K (285° -300° F) and 840 grams per liter (112 ounces per gallon) of the "Ebonol S-34" salts. The processing time was varied from 0.75 to 10 minutes in an effort to obtain the best optical surface characteristics. These results are shown in Figure 10 and 11. Small changes in processing time did not appear to affect the optical surface properties of this oxide coating as much as similar changes did with the copper oxide, although the emittance and solar absorptance curves were somewhat similar. Two minutes appeared to be the optimum processing time with regard to the highest solar absorptivity and lowest thermal emissivity. The spectral reflectance curve in Figure 11 shows a sharp drop near the end of the visible light region (0.7 μ m) but a sharp rise from that point into the infrared, resulting in a solar absorptivity (over the entire region) similar to that of cupric oxide.

Like "Ebonol C," "Ebonol S-34" has moderately good resistance to moisture. Test panels subjected to a high humidity environment for 43 days showed no change in optical properties although a few isolated spots of rust were visible.

"EBONOL Z"

"Ebonol Z" is produced by immersion of zinc in a mild alkaline solution operated at a temperature of $433^{\circ} - 453^{\circ}$ K (160[°] - 180[°]F). Like "Ebonol C" and "S-34," a lustrous, jet black finish can be obtained on a polished surface which is reported to have fair corrosion resistance in a mild atmosphere or indoors. The usual processing time for "Ebonol Z" is from 4 to 8 minutes in a bath containing 45 grams per liter (6 ounces per gallon) of the "Ebonol Z" salts. In this study this concentration was maintained, but the processing time was varied as with the previous "Ebonol" coatings in search of a coating with the best combination of high solar absorptivity and low emissivity. Figure 12 shows the results of this determination. For the most part, the emissivity of this coating was considerably higher than the emissivity of coatings produced in the other "Ebonol" solutions. The emittance of 0.13 for the 2 minute coating was comparably low but was offset by a lowered solar absorptivity at this 2 minute point. This sharp drop in emissivity and solar absorptivity from 1.5 to 2.0 minutes treatment time reflected a considerable deviation from the optical pattern of the coatings produced in "Ebonol C" and "S-34" baths. However, no attempt was made to determine the cause of this deviation in view of the relatively poor overall optical selectivity shown by this coating.

"ELECTROLESS BLACK MAGIC"*

"Electroless Black Magic" produces a black coating on aluminum by simple immersion in a chemical salt mixture. The recommended concentration of this mixture is 4.5 grams per liter (6 ounces per gallon) of the "Black Magic" salts; the operating temperature is $350-355^{\circ}K$ (170-180°F). The coating is reported to be abrasion resistant, attractive, and have nominal corrosion resistance. ⁽⁹⁾ In an effort to optimize the solar absorption properties of this coating, the processing time was varied as with the other black finishes, but the bath was operated at the above stated temperature and concentration. The coating was applied to three different aluminum alloys. The solar absorption properties of the coating produced under these conditions are presented in Figure 13. These results showed that the absorptivity of this coating was moderately high under most of the varying time periods, but the emissivity was also unfavorably high resulting in low $\alpha_{\rm s}/\epsilon$ ratios. The optical properties varied somewhat with processing time, but none of the varying conditions resulted in coatings with good optical selectivity.

ANODIZED COATINGS

DYED ANODIZED ALUMINUM

Anodized coatings on aluminum (aluminum oxide) are produced by the electrolytic reaction of aluminum with the oxygen liberated from a suitable electrolyte, e.g., aqueous solutions of sulfuric acid. The oxide coating lends itself to coloring with dyestuffs by virtue of an extremely porous outer layer of the coating which absorbs the dye.

Panels of aluminum alloy 2024-T3 were colored black by this method and their solar absorption properties evaluated. The anodizing of the aluminum alloy was done in an electrolyte of 15 percent (by weight) sulfuric acid in water at a current density of 12 mA/cm², and a bath temperature of 301° K (82° F). Subsequent blackening was carried out by immersing the anodized

*Trade name of The Mitchell-Bradford Company

aluminum alloy in an aqueous solution of a commercial organic dye (Aluminum Black BK produced by Sandoz Chemical Works, Inc.) operated at a temperature of 339° K (150° F), and a bath concentration of 10 grams per liter (1.3 ounces per gallon). From results of previous optical studies of this coating, conventional anodized thicknesses were known to exhibit high emissivities. In this study, the thickness of the coating was therefore reduced by minimizing the anodizing time in an effort to lower the emissivity. As shown in Figure 14, these test results showed that the emissivity could be lowered somewhat by a reduction in the thickness of the anodized coating but not to an acceptable level, especially since a lowering in solar absorptivity also occurred. In each case, the emittance was higher than the solar absorptance resulting in an α_s / ϵ ratio of less than one.

ANODIZED TITANIUM

Various colored films are produced on titanium by the reaction of this metal with many electrolytes. The color of the film depends upon the electrolyte and several operating variables such as time, temperature, and current density. Various colored films, produced by varying the voltage and time in a 5 percent (by wt.) solution of sodium hydroxide, were evaluated with regard to solar absorption properties. The temperature of the electrolyte was 350[°]K (170[°]F). Although varying the anodizing voltage and time did produce different colored films, it did not produce significant changes in the optical characteristics of the films. The test results are shown in Figure 15. Results of previous studies had shown that thick anodic coatings on titanium exhibit high emissivities, e.g., an emittance of 0.81 results from anodizing 20 minutes in certain acid electrolytes. The anodizing time in this study was therefore minimized in an effort to lower the emissivity. This was accomplished as evidenced by the emittance values of 0.15 to 0.16 shown in Figure 15. However, these values are still relatively high, especially in view of the fairly low solar absorptivity exhibited by the coatings.

CONCLUSIONS

Results of this study showed that the selective solar absorption characteristics of the electroplated coatings and those produced by conversion coating in "Ebonol C" and "Ebonol S-34" baths make them favorable for use as absorbing surfaces in high efficiency flat plate solar collectors. The other coatings studied did not possess a combination of selective optical properties which would make them attractive for such application. These optical results are summarized in Table II. The values listed in this table reflect the optimum α_s and ϵ that could be obtained under the varying experimental conditions of the respective processes, and were determined at ambient temperature by Gier Dunkle Reflectometers.

Black nickel on nickel plated aluminum exhibited the best combination of selective optical properties of any of the coatings studied. With the exception of "Electroblack" which was plated on a substrate of brass, black nickel had ' the highest α_s / ϵ ratio. Black nickel was consistently plated on aluminum heat exchanger panels with an average α_s and ϵ of 0.92 and 0.06, respectively; the α_s / ϵ ratio was 15.3. A serious drawback to black nickel is its high susceptibility to degradation in the presence of high moisture environments. It had the lowest resistance to high humidity of any of the plated coatings investigated.

Both the black chrome coatings exhibited high solar absorptivities. Although the Dupont coating showed the highest of any of the plated coatings, it had the lowest $\alpha_{\rm s}/\epsilon$ ratio due to its relatively high emissivity. It appeared that this coating also had the greatest moisture resistance of any of the coatings studied; those that did not show favorable optical characteristics were not tested for moisture resistance. The "electroblack" coating exhibited the highest $\alpha_{\rm s}/\epsilon$ ratio, but this high ratio resulted only when the coating was plated on a substrate of brass. When it was plated on other substrates such as nickel, the solar absorptivity was lowered which resulted in smaller $\alpha_{\rm s}/\epsilon$ ratios.

TABLE I EFFECT OF SIMULATED SUNLIGHT ON BLACK NICKEL PLATED ALUMINUM

EXPOSURE TIME, ESH*

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OPTICAL PROPERTIES	Before Exposure	2020	2828	6940	8340	8640	1
Solar Absorptance	0.89	0.88	0.85	0.82	0.82	0.83	1
Emittance	0.06	0.05	0.06	0.06	0,07	0.08	
	,						

*Equivalent Sunshine Hours - Lamp intensity equals 0.8 solar constant.

TABLE II SOLAR ABSORPTION PROPERTIES OF VARIOUS COATINGS

Costing	Substrate	Optical Property			
Coating		α _s	é	α _s /ε	
Black Nickel	Nickel Plated Aluminum	0.94	0.06	16	
Black Nickel	Nickel Plated Steel	0.92	0.07	13	
Dupont Black Chrome	Nickel Plated Steel	0.95	0.12	8	
Harshaw Black Chrome	Nickel Plated Steel	0.90	0.07	13	
Electroblack	Brass	0.86	0.05	17	
Black Cupric Oxide (Ebonol C)	Copper	0.84	0.06	14	
Black Ferroferric Oxide (Ebonol S)	Steel	0.86	0.07	12	
Black Zinc (Ebonol Z)	Zinc	0.75	0.13	6	
Chemically Blackened Aluminum (Black Magic)	Aluminum	0.82	0.12	. 7	
Black Dyed Anodize	Aluminum	0.54	0.75	1	
Anodize	Titanium	0.78	0,15	5	



































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