SELECTIVE CONVERSION COATINGS ON NICKEL AND STAINLESS STEEL

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1. Introduction.- Chemical conversion processes offer potential for the production of low-cost selective surfaces at high production rates. Two such surfaces have been developed by Inco Europe Ltd.

The first, a chemically grown oxide film on stainless steel, /1-4/, was developed over the period 1966 to 1972 for decorative finishes, and as such is available in a range of colours. Although the reflectance spectra of several of the colours were given in an early paper /2/ and these demonstrated quite good solar absorptance, combined with high reflectance into the near infrared, the widespread interest in the treatment for solar collectors did not develop until the work of Smith /5/ and others /6/ in Australia, and more recently Karlsson /7/.

Blue coloured stainless steel, blue having the best solar absorptance of the range of colours, has an absorptance value 0.90 combined with a 100° hemispherical emittance of 0.13.

The second surface, produced by a proprietary process on nickel /8-10/, is essentially a thin mixed nickel oxide. It has enhanced solar absorptance (0.97) associated with a topological microroughness, combined with low thermal emittance $E_{h100°C}$ = 0.09. Both surfaces are currently produced on a commercial scale by MPD Technology Ltd. The stainless steel surface treatment, marketed under the name Skysorb [1] is available for all grades of stainless steel in sizes up to 2m x 1m. Finished components can be coloured or, alternatively, sheets of coloured steel can be supplied for subsequent in-house fabrication of components. Essentially, the process is a batch process. The nickel surface, termed Maxorb [1], is available in two forms : Maxorb foil and Maxorb plate.

The Maxorb foil is produced in thickness ranging from 10 to 50 μm. The standard thickness foil, 13 μm, is supplied adhesive backed for application to metallic and non-metallic substrates. Some of the advantages of the concept of foils for the application of selective surfaces were pointed out by Mason /9, 10/ and more recently reviewed by Lampert /11/. Currently, Maxorb is produced in widths up to 148mm, but later this year will be available up to 500mm. Maxorb plate has to date been applied to components up to 3m in length.

In this paper, we shall review the properties of both these selective surfaces and dwell on the latent results, particularly those relating to aspects of durability.

2. Properties of Maxorb.- 2.1. Optical properties and structure.- As described earlier, the solar absorptance of Maxorb is very high, being measured at 0.97 for AM2 spectrum. The repeatability of the process is such that the solar absorptance can be kept within the range 0.96 to 0.98 during a production run of several thousand square metres.

We had previously reported /9/ the thermal emittance of the surface at 100°C as 0.08 to 0.12 depending upon the method of measurement. This difference has been resolved in the work of Pettit /12/ who has shown that the values given by the Willey emissometer have to be corrected to correlate with other measurement methods.

Table I.- Total hemispherical emittance of Maxorb solar foil

Temperature °C	Hemispherical Emissivity ε_h	Measurement Method	Source
29.3	0.065	Calorimetric	Beens 13
47.8	0.071	"	"
97.4	0.079	"	"
120.7	0.088	"	"
151.4	0.098	"	"
100	0.08	Corrected Willey Emission	
100	0.07	Gier Dunkle Emissometer	Mason 9

Table I lists values of total hemispherical emittance for Maxorb foil measured calorimetrically by W.W. Beens *et al.* /13/. Also included are

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corrected Willey results and values obtained with a Gier Dunkle emissometer. Agreement is very good.

2.2. Durability of the surface.- Maxorb has been subjected to a wide variety of tests to establish its resistance to degradation under conditions prevalent to flat plate collectors and under conditions which are considered to accelerate the degradation processes. Some of the tests have been performed by Masters /14/ as part of a much wider programme, aimed at the development of standards for evaluating solar absorber materials. Some tests were performed in-house.

The surface optical properties, measured after ageing in air for various periods of time up to 300°C, are listed in table II. There is no significant change in properties over the conditions examined.

Table II.- Effect or air ageing on the optical properties of Maxorb

Time/Days	AGEING TEMPERATURE							
	150°C		200°C		250°C		300°C	
	α_s	ε_{h100}	α_s	ε_{h100}	α_s	ε_{h100}	α_s	ε_{h100}
0	0.98(+)	0.09						
10			0.98	0.09			0.99	0.09
84			0.98(+)	0.10	0.98	0.08		
98	0.97(+)	0.08						
200			0.97	0.07				
463			0.98	0.09				

(+) Masters /14/

Heat exposure for shorter periods of time, up to 1 hour, suggest that the surface is stable in air up to 400°C /9/. Clearly, the surface has adequate temperature stability for use in single or double glazed collectors.

Moisture resistance was determined using both cyclic (to MIL STD 810B) and steady state testing. In view of the accelerated nature of these tests, there was surprisingly little change in absorptance (see Table III).

Although the results indicate a high degree of stability, their significance can only be assessed in relation to the equivalence between such tests and real time exposure. Unfortunately, since Maxorb has only been produced for 3 years - no long time results are available.

Table III.- Effect of moisture on solar properties of Maxorb solar foil

Test Conditions	Time Period (Days)	Optical Properties	
		α_s	$\varepsilon_{h100°C}$
Cyclic			
Mil-Std-810B	8	0.97	0.08
	35	0.96	0.08
	138	0.96	0.07
Steady State			
92°C 97% R.H.	0	0.98	0.09
	147(+)	0.98	0.09
40°C 100% R.H.	42(+)	0.99	0.09
	84(+)	0.95	0.08

(+) Results due to Masters /14/

In our tests, samples have shown no measurable change in surface properties after stagnating in a single-glazed insulated box on our laboratory roof for 2 years. Similar stagnation tests, to date for a shorter period, have been reported by Masters /14/ and are reproduced in table IV. These tests are continuing.

Table IV.- Stagnation tests at test sites in the U.S.A.

Solar Properties

Surface	Initial values		Test site					
			NBS		Arizona		Miami	
			253 days		128 days		124 days	
	α_s	ε_{h100}	α_s	ε_{h100}	α_s	ε_h	α_s	ε_h
Maxorb Foil on Al	0.98	0.09	0.98	0.09	0.98	0.09	0.98	0.08

2.3. Stability of adhesive.- Our previous work /9, 10/ described how a silicone-based pressure sensitive adhesive had been elected for fixing the foil to substrates because of its combination of temperature stability and weathering resistance. In order to assess the degree to which the foil is bonded to the substrate, peel tests (Method ASTM D1000) were performed.

The results of temperatures up to 200°C on the peel strength of Maxorb on Al are illustrated in figure 1. These results are for a 90° peel mode. The performance of DC 282 (produced by Dow Corning) the adhesive initially selected for Maxorb foil, shows a fall off in properties after an initial cure onto the substrate. In spite of this, there was no sign of edge delamination even after the most severe conditions (200 days at 200°C).

More recently, a silicone with heat stabilization SR 6574 (General Electric Silicones) has been evaluated. The results (see Fig. 1) show a significant improvement over DC 282.

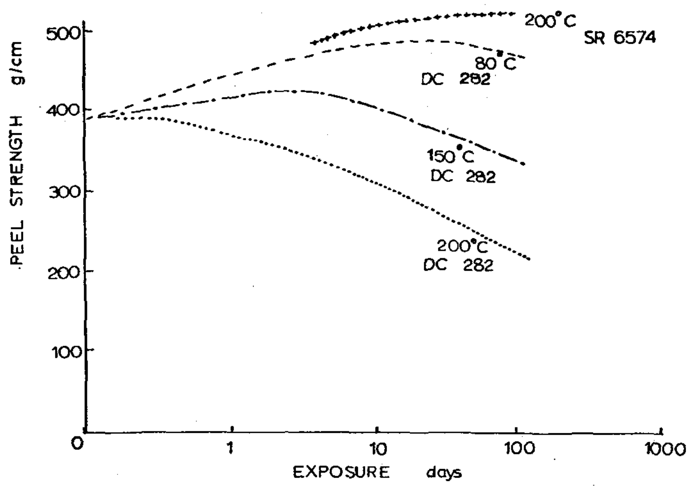

Fig. 1.- Effect of temperature on the adhesion of Maxorb to aluminium for two adhesives DC 282 and SR 6574.

The above results relate to aluminium as a substrate. Similar tests have been made for copper and mild steel substrates.

The behaviour with mild steel is virtually identical to aluminium. In the case of copper, however, there is evidence of delamination after exposure at 200°C. This appears to be due to oxidation of the copper under the adhesive which clearly commences at the edge of the foil.

The foil also acts as a humidity barrier and protects the underlying substrate. This is particularly evident upon examination of Maxorb foil coated Al and mild steel samples after cyclic humidity tests.

On removal of the foil, a clear cut-off can be seen between the protected and unprotected areas. Some indication of the effect of cyclic humidity temperature tests on the adhesion of the two adhesives examined can be seen from figure 2. Under the conditions of MIL STD 810B, temperatures up to 71°C continued with high humidities (95% R.H.), there is no significant change in adhesion after 100 days. These tests are continuing.

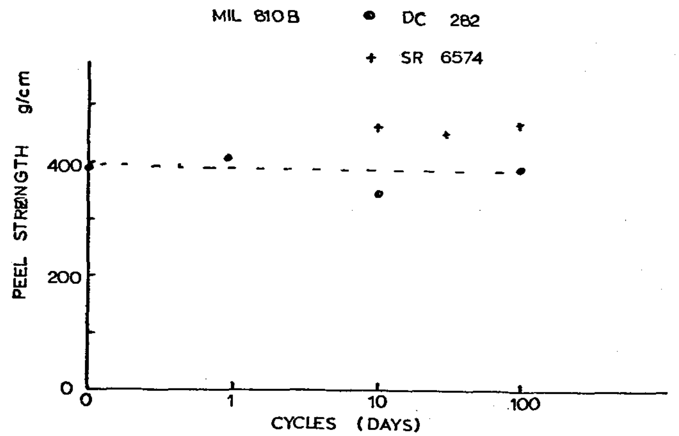

Fig. 2.- Effect of humidity cycling on the adhesion of Maxorb to aluminium

2.4. Adhesion to non-metals.- As well as its application to metals in flat-plate solar collector water heaters, Maxorb solar foil may be considered for use in passive systems. A recent theoretical analysis by J.S. Van Wieringen /15/ showed that double glazing is necessary with neutral black-painted walls in order to get reasonable output. However, a single glazed wall with a selective surface gives increased yield, because of better efficiency and a reduction in nocturnal losses, and holds out the promise of lower cost.

Preliminary experiments, aimed at assessing the adhesion of Maxorb, with its normal P.S.A. adhesive backing (DC 282) to common construction materials such as brick and concrete showed that there was little adhesion, due to the uneven and/or friable nature of these surfaces. In order to stabilise the surface, various pre-treatments were examined.

The most successful treatment, one meeting the requirement of temperature stability together with room temperature cure in air, was a silicone resin produced by Dow Corning designated R-4-3117 Conformal Coating. Peel strengths after up to 10 days at 150°C are given in table V.

Table V.- Adhesion of Maxorb (DC282) to common building materials

Substrate	Coatings 1	2	3	Pre-test Exposure	Peel Strength g/cm
Concrete	Cement render	Tetrion	R-4-3117	As applied	178
"	"	"	"	24h/150°C	337
"	"	"	"	4 days 150°C	380
"	"	"	"	10 days 150°C	380
Brick	R-4-3117	-	-	As applied	178
"	"	-	-	24h/150°C	315
"	"	-	-	4 days 150°C	315

Concrete, having an inherent absorbent irregular surface, called for the application of successive layers of materials having progressively smoother surfaces. These multilayer specimens, with a R-4-3117 finish realized the highest peel strengths, typically 380g/cm.

Surfaces coated with a standard finishing plaster i.e. Thistle Finish, suffered from spalling after exposure to 150°C. Since this was used for the accelerated tests the plaster was replaced by Tetrion filler (manufactured by Tetrosyl Ltd.) which proved stable at this temperature.

Brick, having a lower absorptivity to liquids and generally a smoother surface than concrete responded to simple surfacing routes. Results from a single layer of R-4-3117 applied directly to the brick surface were only about 20 % lower than those obtained from a three layer treatment of concrete. The results supports the premise that Maxorb can be applied fairly readily to brick or concrete surfaces.

2.5. Surface protection.- Like all high absorptance selective surfaces Maxorb is prone to finger marking. It is not possible to remove the mark subsequently. Although the effect on surface properties is minimal, changing the absorptivity from 0.98 to 0.97 and having no measurable effect on emittance, such marking is unsightly.

Attempts to improve handleability focussed on overlaying the black surface with a thin plastic layer. A Dow Corning silicone resin, DC 808, displays a suitable combination of properties, namely, good heat resistance combined with good flexibility in the cured condition. A three roll reverse coating technique on a commercial roller coated combined with dilution of the resin with up to 90 % solvent enabled the application of controlled thicknesses down to about 0.1 µm.

The effect of resin thickness on optical properties is illustrated in figure 3. For use in flat plate collectors an emittance of 0.15, corresponding to a resin film thickness of 0.5 µm, would be quite satisfactory. This surface layer would provide added security against possible degradation, the only disadvantage being the reduced solar absorptivity (0.935).

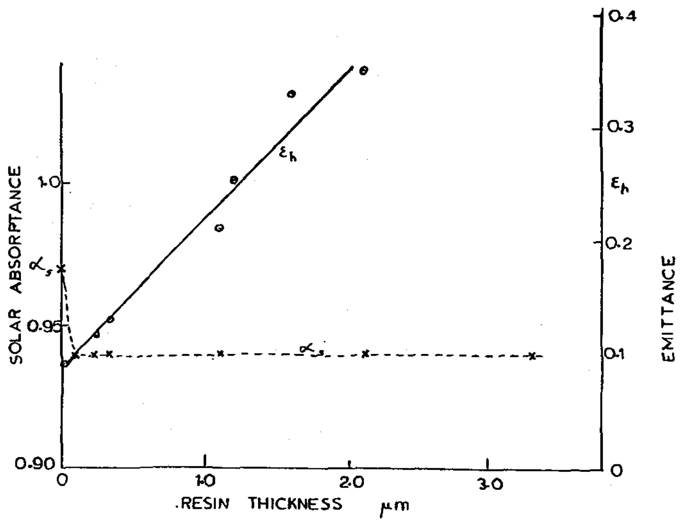

Fig. 3.- Optical properties of Maxorb with DC 808 front coating

2.6. Maxorb plating.- There are three main areas where Maxorb foil may not be used, in concentrating collectors where the higher temperatures prevent the use of adhesives, in vacuum tubes where degassing of the adhesive can cause problems, and in flat plate collectors having profiled surfaces unsuitable for laying foil. In all three cases the component can be nickel plated and then blackened using the same process as used for foil. In general, the solar properties of the blackened plate are very similar to the foil, typical values being α = 0.96, $\varepsilon_{h100°C}$ = 0.12.

3. Coloured stainless steel.- The Inco colouring process produces an oxide film on the surface of the steel which is basically colourless and transparent. As the film-thickness increases a range of interference colours are obtained in the order bronze, blue, gold, purple and green /3/.

As formed these films are soft and porous. An electrolytic treatment is used to seal the films. This treatment confers wear and scratch resistance on the films and also improves their handleability, permitting them to be degreased and cleansed without detriment to their appearance.

3.1. Optical properties.- These films have been shown to be solar selective /2, 5/.

Values of solar absorptance obtained on interference films using relatively simple laboratory techniques, such as those developed for assessing black surfaces, are of doubtful validity. The reasons for this have been discussed by Mason /16/.

Values of solar absorptance of 0.90 - 0.95 have been obtained in the Skysorb blue range of colours using these techniques, which is in agreement with figures quoted generally by others working on similar films. However, it is felt that these can be misleading and their use should be discouraged.

An alternative method of estimating solar absorptance and one which is perhaps of greater practical significance, is by use of efficiency curves. Using an ASHRAE 93-77 standard procedure the efficiency curves were determined for two identical collectors fabricated from A.I.S.I. Type 304 annealed stainless steel, one coated with Skysorb selective surface, the other painted matt black. (Nextel 101-C10 Black Velvet)

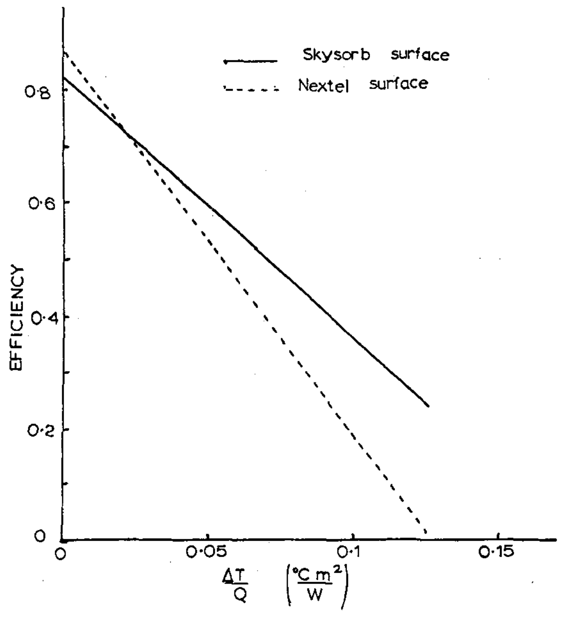

Fig. 4.- Performance graph of two stainless steel collectors

The performance curves obtained are shown in figure 4. The efficiencies of the collectors are expressed as

Skysorb surface $\quad \eta = 0.823 - 4.65 \frac{\Delta T}{Q}$

and

Nextel surface $\eta = 0.869 - 6.86 \frac{\Delta T}{Q}$

The ratio of the two maximum efficiency values is the ratio of the respective solar absorptances.

$$\frac{\alpha \text{ Nextel}}{\alpha \text{ Skysorb}} = \frac{0.869}{0.823}$$

Taking α Nextel as 0.97 this gives α Skysorb as 0.92. This is in reasonable agreement with values calculated for similar films by Smith /5/ from reflectance spectra using the A.M.2 solar spectrum.

The thermal emittance at 100°C, E_{h100}, measured using a Willey emissometer did not vary over the range of blue colours obtained by the Inco process. Thicker films i.e., purple and green, gave total hemispherical emissivities at 100°C of 0.12 - 0.15.

Auger spectra were taken on two samples of A.I.S.I. Type 304 steel representing the bronze/blue border and blue/gold border, that is either end of the blue range obtained by the Inco process. These were sputtered to give semi-quantitative chemical analysis of elements as a function of depth.

Table VI.- Auger electron spectroscope. Depth profile analysis of coloured stainless steel, Type 304.

Sample	Approx Thickness µm	COMPOSITION Wt %					
		S	C	O	Cr	Fe	Ni
Bronze/Blue	0	6.7	2.8	36.0	26.9	19.6	7.6
	0.01	0.9	0.3	28.4	23.8	32.0	13.8
	0.02	0.8	0.3	24.7	22.5	36.7	14.4
	0.03	0.6	0.2	18.3	17.9	48.4	14.0
	0.04	0.5	0.2	13.1		58.7	13.3
	0.05	0.3	0.2	8.0		69.5	12.4
	0.06	0.2	0.2	5.8		73.6	11.8
	0.07	0.0	0.2	3.0		77.8	10.8
	0.10	0.0	0.3	0.0		80.7	9.5
	0.14	0.0	0.3	0.0		79.6	9.5
Blue/Gold	0	7.3	1.6	39.1	27.3	17.7	6.3
	0.01	1.1	0.2	36.2	30.6	19.8	11.4
	0.03	1.2	0.0	33.0	29.6	22.2	13.4
	0.05	0.9	0.0	30.2	26.2	26.5	15.5
	0.07	0.7	0.1	22.1	19.3	41.7	15.7
	0.09	0.4	0.2	10.7		62.8	12.9
	0.11	0.1	0.3	2.4		76.5	10.3
	0.14	0.0	0.4	0.0		79.3	9.5

The results, table VI, indicate that the oxide is rich in chromium compared with the substrate steel, but also contains iron and nickel. The oxide thickness over this colour range varies from approximately 0.09 µm to 0.13 µm.

3.2. Durability of Skysorb surfaced stainless steel.- 3.2.1. Atmospheric corrosion tests.- Corrosion tests have been made at a number of industrial and urban sites on Skysorb surfaced stainless steels over periods extending to 13 years for samples without the hardening treatment and eight years for samples with the hardening treatment. Generally, samples were exposed on south facing racks at 45° to the horizontal and were unprotected. No attempt was made to stimulate flat plate collector test environment.

There was no evidence of colour change in any of the specimen compared with unexposed controls. A.I.S.I. Type 304 and Type 430 steels were exposed. The corrosion resistance of the coloured and hardened samples of both types of steel was generally superior to that of untreated samples.

Skysorb surfaced A.I.S.I. Type 304 steel samples have also been exposed at Marine test sites. Panels facing seawards were placed 250 m from the ocean. All samples were unprotected.

Table VII.- Exposure of Skysorb surfaced stainless steel to marine atmospheres

Material	A.S.T.M. Corrosion Rating 2 . 5 years Exposure
Skysorb surface on Type 304 L	9
"	9
"	8
"	9
A.I.S.I. 304 L	6
A.I.S.I. 304 L	5

A.S.T.M. Rating

10	No defective area	7	0.5% defective area
9	0.1% defective area	6	1.0% defective area
8	0.25% defective area	5	2.5% defective area

Colour retention after 2 .5 years' exposure was very good. Only a very slight difference was detected when comparing exposed panels with unexposed controls.

A.S.T.M. corrosion rating data on the marine atmosphere corrosion test samples is given in table VII. Skysorb surfaced samples showed only minimum corrosion spotting and were superior to untreated samples.

3.2.2. Accelerated corrosion tests.- The general trends of the atmospheric corrosion tests were reproduced in accelerated testing conditions.

CASS Tests were made on A.I.S.I. Type 304 and Type 430 steels with and without Skysorb surfaces according to procedures specified in BS4601, 1970. After 24h exposure, the Skysorb surfaced panels showed less rust stain than the untreated panels. Blue coloured but unhardened panels were inferior to Skysorb surfaced panels.

Cyclic tests in an atmosphere containing 500ppm of sulphur dioxide were made on Skysorb surfaced Type 304 steel. The moisture content of the atmosphere was greater than 80% and test temperatures varied in the range 24-48°C. After 200 cycles, each of one hour duration, there was no change of appearance of the Skysorb surfaced panels. Untreated A.I.S.I. Type 304 steel showed some corrosion.

Results of pitting corrosion tests in a solution of 10% ferric chloride for 1 hour at 20°C are presented in table VIII.

Table VIII.- Corrosion tests on Type 304 stainless steels in 10% ferric chloride solution at room temperature for one hour.

Specimen Condition	Surface Finish	Appearance	Pit damage N/cm^2	Remarks
Uncoloured	Mirror Polish	Moderate Pitting	14.0	Large & fine pits
Blue unhardened	"	Moderate/heavy pitting	7.0	Large deep pits
Skysorb hardened	"	Very slight pitting	$<$ 1.0	One or two isolated small pits
Uncoloured	2B	Very heavy pitting	35.0	Large & fine pits
Skysorb (Hardened)		Very slight pitting	5.0	Very fine & shallow pits

The Skysorb surfaced steel showed superior resistance to attack than the coloured but unhardened or the untreated steel.

3.2.3. Stagnation tests.- Skysorb coated samples of stainless steel A.I.S.I. Type 304 with both mirror polished and bright annealed surfaces and A.I.S.I. Type 444 steel mirror polished were exposed in single glazed, back-insulated boxes on our Birmingham Laboratory roof. The vented boxes allowed condensation and the natural change in air. Comparison with unexposed control samples showed no change in colour and no evidence of rust spotting after 1.5 years exposure. These tests are continuing.

3.2.4. Thermal stability.- Tests have been made to determine the effect of heat on the colour stability, adhesion and appearance of the coloured film.

Exposure of the coloured and hardened steel to boiling water for 20 days showed no change in any of these properties.

Exposure in a dry atmosphere at 200°C for 20 days, showed a slight alteration in shade during the early stages of exposure. It is thought that this is due to the removal of water present in the film.

Indications of colour change for longer exposure tests are listed in table IX.

Table IX.- Effect of long term thermal exposure on colour stability of stainless steels.

Steel Type	Temperature	As coloured & hardened	Period of Exposure	
			after 56 days	after 112 days
304	150°C	Blue	Blue somewhat paler	Blue
444	150°C	Blue	Blue	Paler blue
17 Cr	150°C	Blue	Light blue	Blue/Gold
304	200°C	Blue	Lighter blue	Blue/Gold
444	200°C	Blue	Blue	Very light blue
17 Cr	200°C	Blue	Blue/Gold	Gold

The colour changes for these longer times are dictated by the growth rate of the oxide film. This process depends upon the intrinsic oxidation resistance of the substrate steel as well as the temperature and time of exposure. Of the three types of steel examined, the oxide stability of the simple ferritic steel (17%Cr) is lowest, the austenitic stainless steel, (Type 304) ranks next, whilst Type 444 (containing 18Cr 2Mo) has the greatest resistance to oxide growth at the test temperatures used. Because of the flat maximum in solar absorptance, over the whole range of blue colours, the colour changes produced by up to 112 days at 150°C for Types 304 and 444 steels have little effect on solar absorptance. The change in appearance, and therefore, reduction in solar absorptance for the simple 17% Cr. Steel after 112 days at 150°C probably precludes it as a material for flat plate solar collectors.

The film growth process is more rapid at 200°C. All the steels tested showed some colour instability at this test temperature.

Variation in solar absorptance for mirror polished blue stainless steel A.I.S.I. Type 304 exposed for 1h at temperatures up to 500°C are shown in figure 5. Solar absorptance was calculated from total reflectance spectra. Mirror polished samples were chosen because they offered a more uniform starting surface. These indicated that short time, i.e., minutes, exposure at temperatures up to 300°C is not detrimental to the solar properties of the surface.

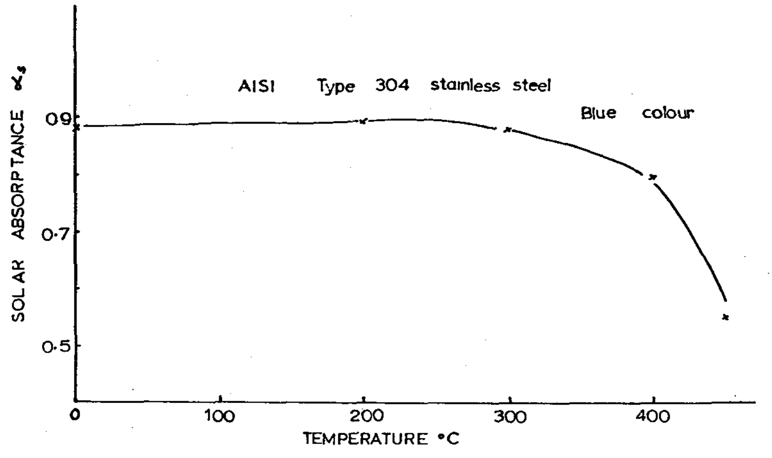

Fig. 5.- One hour temperature exposure on absorptance of blue stainless steel

The results of thermal stability tests indicate that Skysorb coated A.I.S.I. Type 304 and Type 444 have satisfactory thermal stability for single glazed flat plate solar collectors. It is likely that Type 316 austenitic steel would have better colour retention than Type 304 and probably be similar to Type 444.

Although this aspect of film growth precludes use of Skysorb surfaces for solar collectors likely to experience service conditions involving long periods of exposure in air at temperatures above 200°C i.e. concentrating collectors, it does not preclude their use for such collectors where the surface is protected by vacuum.

3.3. Formability.- The adherence of the oxide film has been assessed by subjecting Skysorb coated steels to various commercial cold forming processes.

Coloured sheets up to 1200mm by 900mm have been roll patterned using a process that results in no overall change in dimensions of the sheet and produces local reductions in thickness of up to 30%. Similar sheets have been deformed using roll forming and conventional pressing techniques.

In none of these tests was there any evidence that the film had cracked or become detached from the underlying metal. Samples have been bent through sharp angles and undergone laboratory deep drawing tests with similar results.

A clear advantage of this surface over other reflective surfaces is, therefore, its suitability to a wide range of cold forming methods after colouring.

4. Conclusion.- This paper has reviewed the salient features of two selective surfaces, one on nickel ; the other on stainless steels, which are both now commercially available. Both surfaces have differing advantages which make them complementary rather than competitive in the market place. Skysorb surfaced stainless steel, which has even better atmospheric corrosion resistance than conventional stainless steel, is ideally suited to lower temperature applications. In its present form, it is not recommended for use in non-evacuated concentrated collectors. The surface has excellent formability and handleability allowing the fabricator plenty of scope in maximising its design potential. This colouring process is a batch process.

The Maxorb surface on nickel is a high performance selective absorber which either as a foil or plated component finish can be applied to all substrates. As indicated previously, the blackening can either be a batch or continuous process. In its continuous form, it allows a greater degree of control and greater flexibility in product form. The surface does not have as good atmospheric corrosion resistance as coloured stainless steel but possesses better thermal stability. The cost of both processes is very similar.

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