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Mechanical properties of anodized coatings on 5657 alloy have been studied. Stresses were measured after each process step and after exposure to different humidities and temperatures. Stress is tensile after anodizing and compressive after sealing. Prolonged exposure to low humidity causes the stress to become strongly tensile; the effect of humidity on stress is reversible. Oxide cracking due to differential thermal expansion depends on environmental and metallurgical factors. Plastic deformation of metal occurs under the oxide cracks.

## I. INTRODUCTION

This work is part of a program to obtain information about properties of anodized aluminum coatings that are important to its application as a thermal control surface in a space environment. Since there is no atmosphere in space to support conduction or convection, heat rejection by radiation is the only means to achieve thermal management. Anodic oxide coatings have the high emissivity and low absorptivity that is required for good thermal control. The optical properties are stable in the intense ultraviolet radiation, atomic oxygen, and vacuum of the low earth orbit environment (1). Because of superior optical properties, sulfuric acid anodized 5657 aluminum alloy has been selected as a candidate material for use on Space Station Freedom for certain radiator surfaces (2). Anodic coatings also will be used for thermal control on external structural alloy surfaces.

A potential problem is that anodic coatings may crack under some conditions at elevated temperature. There is concern that extensive cracking of the radiator coating could impair optical properties and reduce heat rejection efficiency. Thermal cracking occurs because the coefficient of thermal expansion of aluminum is about five times greater than that of aluminum oxide. If metal and oxide were each free to expand at some elevated temperature, the metal expansion would be five times that of the oxide. The intimate bond at the metal/coating interface prevents this equilibrium state from being achieved; the metal is constrained to expand less and the oxide is forced to expand more. At a sufficiently high temperature the resulting tensile stress in the coating is large enough to crack the oxide. At low temperatures the forces are reversed and the oxide is placed in compression by metal contraction.

Radiator panels are fabricated by adhesively bonding the anodized sheets to a honeycomb core. This operation is performed at a temperature between 250 and 350°F (120-175°C), depending upon the adhesive used for bonding. Tensile stress in the coating may become large enough at these temperatures to cause cracking.

Expansion and contraction from temperature cycling may result in thermal fatigue. Over the projected 30-year operating life of Space Station Freedom there will be 175,000 orbital cycles, and during each cycle the space station passes in and out of the sun's radiation. During an operational cycle the temperature extremes of the radiator surface are likely to be about +10° to +75°F (-12° to +24°C). Normal operation will be interrupted from time to time, and then a "deep" cycle will occur, during which temperatures may range between -135°F to +120°F (-93° to +49°C). The deep cycle frequency will be between 0.1

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and 1%.

Several years ago, NASA initiated a project with the overall goal to specify process conditions that will produce anodized radiator panels that withstand fabrication without cracking, and for which there is a high confidence of 30-year operating life with acceptable optical properties. The major parts of our involvement with this program are as follows:

- a. Determine coating physical properties (elastic modulus, coefficient of thermal expansion).
- b. Measure coating stress for different process and environmental conditions.
- c. Characterize the effects of coating, metal, and environment on oxide cracking.
- d. Develop thermal fatigue models to predict coating performance in low earth orbit.

In this paper we present observations of coating stress and cracking that may be of general interest, here in our earthbound environment.

## II. EXPERIMENTAL

Aluminum specimens were anodized and sealed, and then exposed to various controlled temperature and humidity atmospheres. Coating stress was measured after each process step, and also during environmental exposure. In separate experiments, cracking of the coating induced by elevated temperature was studied. This was characterized by  $T_c$ , the temperature at which cracks first appeared, and also by the crack density at temperatures above  $T_c$ .

Most results were obtained with 5657 alloy sheet. Stress measurements were made using strips of 10 mil thick H28 temper sheet 0.25" wide x 8" long. Behavior under thermal strain to induce cracking was studied using 2"x2" coupons cut from 25 mil H242 temper sheet. Some reference is made to results with other alloys.

The process sequence was chemical polish → anodize → seal. The strips for stress measurement were coated on one side with a polymer masking compound after the chemical polish. The polishing solution was a mixture of concentrated nitric and phosphoric acids (Bright-Dip) used at 97°C, with an immersion time of 20-30 sec. Anodizing was in 20%  $H_2SO_4$  at 22°C and 15.0V. The average current density was 15.2 mA/cm<sup>2</sup> (14.2 A/ft<sup>2</sup>). Anodizing time was varied to cover a thickness range from 0.3 to 1.2 mil, with most coatings either a nominal 0.5 or 1.0 mil. Sealing was in hot water for 5 min or 20 min. The 2"x2" coupons were sealed at 95°C but, to simplify temperature control during the stress experiments, those strips were sealed in boiling water.

### A. Stress Determination

Stress was determined using beam bending analysis. An Al strip is clamped at one end and anodized on one side. Stress in the coating causes the beam to bend - concave if the stress is tensile and convex if compressive. The magnitude of the stress is proportional to the deflection. The average stress in the coating is calculated using Eq. [1]:

$$\sigma_c^{bent} = \left( \frac{E_s}{1-\nu_s} \right) \left( \frac{t_s}{3l^2m(1+m)} \right) \delta \quad [1]$$

where  $m=t_c/t_s$  and subscripts c and s stand for coating and metal substrate, respectively. E is the elastic modulus and v is the Poisson's ratio, t is thickness, l is anodized length,  $\delta$  is the tip deflection of the strip (reference point: zero stress position). The term  $E/(1-v)$  is the biaxial modulus, and must be used when the width of the strip is greater than its thickness. A more complete form of Eq. [1] is given in ref. 3.

To measure process stress the specimen was suspended vertically with its upper end clamped and the lower end free to move in response to stress changes. The specimen was processed by first raising the anodizing bath to immerse the strip, performing the anodization, then lowering that bath, rinsing and allowing the strip to air dry. Then the seal bath was raised and lowered in the same way. Initially, and after each step, the position of the specimen tip was measured by a laser beam scanning horizontally in the plane of the tip. Deflections were typically of the order tenths of an inch. The length and thickness values needed for the stress calculation were measured directly.

To measure stress during long term exposure, the specimen was transferred to a horizontal clamp attached to a plate on which there was a scale to measure the tip position visually. The plate was placed in a glass desiccator in which a controlled humidity could be maintained by placing a selected desiccant in the chamber. Indicated humidities were either measured with a hygrometer or are nominal handbook values, and are given in ppm (v/v) water vapor. For measurements at temperatures above ambient, the desiccator was placed in an oven. In one experiment, coating stress was measured at  $10^{-6}$  torr in a vacuum chamber with a viewing port.

### B. Coating Cracking

The 2"x2" coupons for testing at elevated temperature were anodized on both surfaces, so no deflection occurred during heating. Stress at temperature was calculated using Eq. [2]:

$$\Delta\sigma_c^{unbent} = \frac{E_s(\alpha_s - \alpha_c) \Delta T}{(1-v_s) \left(2m + \frac{1}{n}\right)} \quad [2]$$

where  $\alpha$ =coefficient of thermal expansion,  $n=[E_c(1-v_s)]/[E_s(1-v_c)]$ , and the other symbols are as defined for Eq. [1]. The value for  $\alpha_c$ , obtained in separate experiments, was found to be close to that for bulk alumina (4). Values for  $E_c$  were calculated from  $\sigma$  vs T measurements with one-side anodized strips (4).

Baseline  $T_c$  values were obtained within 2 days after anodizing by heating coupons in ambient air. Between about 100 and 200°C, temperature was raised in 10°C intervals, the coupons were held for at least 10 min at each temperature, and examined visually for cracks after each step. The test was limited to 200°C because at that temperature the metal deformed. Measurements were made on sets of three duplicate coupons. Other experiments were done in which coupons were placed in controlled atmosphere after the anodizing process, and subsequent heating was either in this same atmosphere or in ambient. Although the range in  $T_c$  for a particular coating/alloy was rather broad, at least  $\pm 15^\circ\text{C}$  in replicate experiments, it served as a general indicator of cracking tendencies with a possible link to cracking during long-term thermal cycling (3).

To determine the effect of thermal cycling, coupons were hung from a chain that moved between heated and cooled chambers in a sealed enclosure. The

humidity was kept at a low level by maintaining a flow of dry air (175ppm) or dry nitrogen (50-80 ppm) through the enclosure. Temperature on the hot side was controlled to within 0.2°C. Coupons were viewed for crack observation through a window located in a short buffer zone between the hot and cold sides. Each cycle took 9.4 minutes.

### III. RESULTS AND DISCUSSION

#### A. Stresses

1. Process Stress - Fig. 1 shows typical values for the stress in nominal 0.5 mil coatings with 20 min seal at each step in their history. The stress after anodizing ( $\sigma_a$ ) does not depend on coating thickness; there is some dependence on process conditions and particular alloy, but  $\sigma_a$  is always tensile and relatively small. The stress after sealing ( $\sigma_s$ ) is always compressive and increases with seal time. There is a dependence on the ratio of coating to substrate thickness ( $m$ ), with  $\sigma_s$  most compressive for small values of  $m$ . In our experiments,  $\sigma_s$  ranged from -3 to -8 ksi.

2. Effect of Humidity - The ambient humidity has a profound effect on coating stress, as illustrated in Fig. 1. The stress shifts from compressive to tensile when the humidity drops below about 2000 ppm (9%RH at 25°C). The tensile stress increases as humidity drops, and reaches its highest value of 16 ksi at  $10^{-6}$  torr vacuum. In dry atmospheres there is little dependence of  $\sigma$  on coating thickness or seal time. The stress increase is accompanied by a

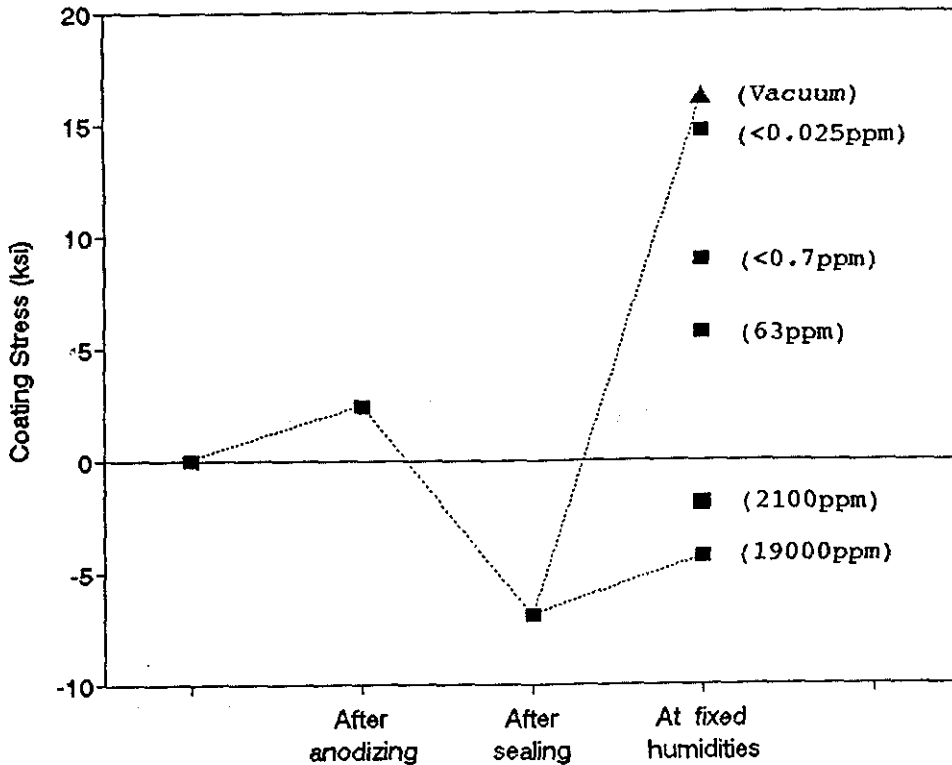


Figure 1. Stress history of 0.57 mil oxide coatings with 20 min seal.

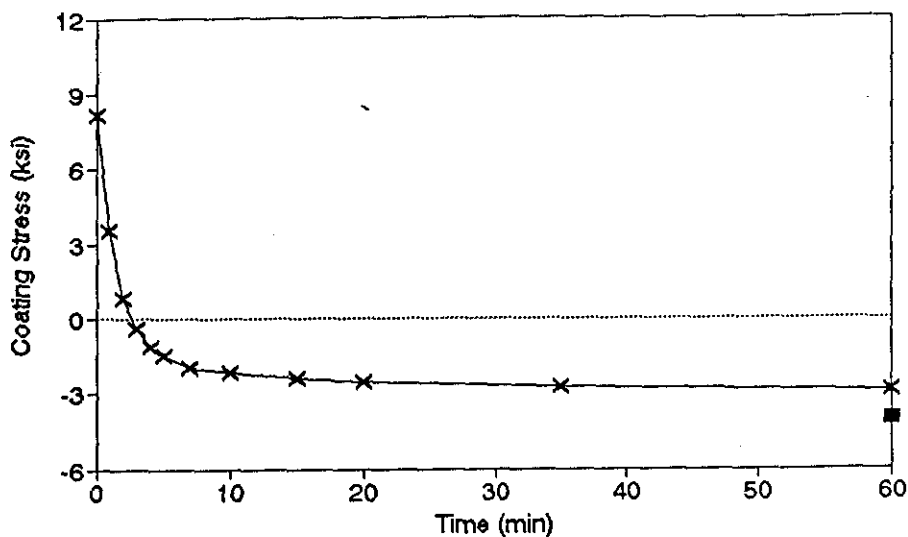


Figure 2. Stress vs time after re-exposure to ambient humidity from 0.7 ppm humidity. Coating is 1 mil thick with 20 min seal. Final stress measured after 149 hours in ambient humidity is shown as filled squared (■).

loss of coating weight, presumably loss of water. For example, for a 0.9mil/20min seal coating stored over  $P_2O_5$  (humidity < 0.025ppm),  $\sigma = 14$  ksi, an increase of 16-18 ksi from ambient humidity, and the weight loss corresponds to 1.1% of coating weight (10% of total water content).

The change in stress with humidity is reversible. Fig. 2 shows that upon re-exposure to ambient humidity from 0.7 ppm, the stress becomes compressive within several minutes and the original value is restored within hours.

3. Effect of Temperature - Fig. 3 shows a typical measurement of specimen deflection vs temperature, measured at ambient humidity. The temperature was raised in steps of 3-5°C and held for 1-1.5 hr at each step. The same result is obtained by heating at an approximately steady rate of about 4-5°C/hr. Cooling to room temperature was not controlled, and most of the temperature drop occurred within a few hours. From Eq. [1], stress is proportional to deflection, so it follows from these measurements that the stress increase is proportional to temperature, and the stress change is reversible upon decreasing the temperature. This behavior was observed up to 70°C, the highest temperature used in these measurements. It was found that the temperature dependence of stress,  $\Delta\sigma/\Delta T$ , is smaller at low humidity than at ambient humidity.

The elastic modulus of the coating ( $E_c$ ) can be calculated from  $\Delta\sigma/\Delta T$ . (4). The modulus is found to be independent of coating thickness and seal time. At humidities of 590 ppm and higher,  $E_c$  appears to be constant at an average value of 19,700 ksi. At humidities of 63 ppm and lower, the average modulus is 11,200 ksi.

#### B. Cracking with Step-wise Heating

With 25 mil 5657 sheet, the average baseline  $T_c$  value is 132°C for 0.5 mil oxide, and 116°C for 1 mil oxide, with a range of about  $\pm 15^\circ C$  for each. No

dependence of  $T_c$  on seal time was found. Using Eq. [2], the corresponding ranges for failure stress are 35 to 49 ksi for 0.5 mil and 26 to 39 ksi for 1 mil oxide. It was generally found that thicker coatings cracked at lower stresses.

Cracks commonly were in the rolling direction and extended across the coupon. Visually the cracks appeared either linear or smoothly curved, but at low magnification, say 100x, they were seen to undulate in response to local variation in stress and/or material properties. It was not uncommon for cracks to propagate in response to an evident local defect, and sometimes in a non-rolling direction. An existing crack stopped the advance of a new crack; cracks never crossed. Cracks that developed between 120° and 190°C were 1-2  $\mu\text{m}$  wide.

Long term exposure at different humidities - Anodized coupons were stored for 4-5 weeks in different environmental conditions and then tested for cracking. These tests are summarized in Table 1. After storage, the first three sets of coupons listed were returned to ambient atmosphere for stepwise heating. Long term dry storage (2-5 ppm) at moderate temperatures has no effect on cracking;  $T_c$  and crack density are the same as for the baseline condition. It was shown earlier that dry storage increases coating tensile stress, but that after returning to ambient the stress quickly returns to the usual compressive value. Remaining at the higher stress level for extended time apparently has no permanent effect on the oxide. On the other hand, cracking occurs at a lower temperature and with increasing density as storage humidity is increased. Slow reaction between the oxide and water vapor apparently degrade oxide mechanical properties.

The last two data sets in Table 1 show results for coupons stored in dry atmosphere at elevated temperatures. Cracks were seen in the coatings when they were removed from the test chamber and they were not subjected to further step-wise heating. It appears that at elevated temperature a slow change occurs in the coating at low humidity that can initiate cracks.

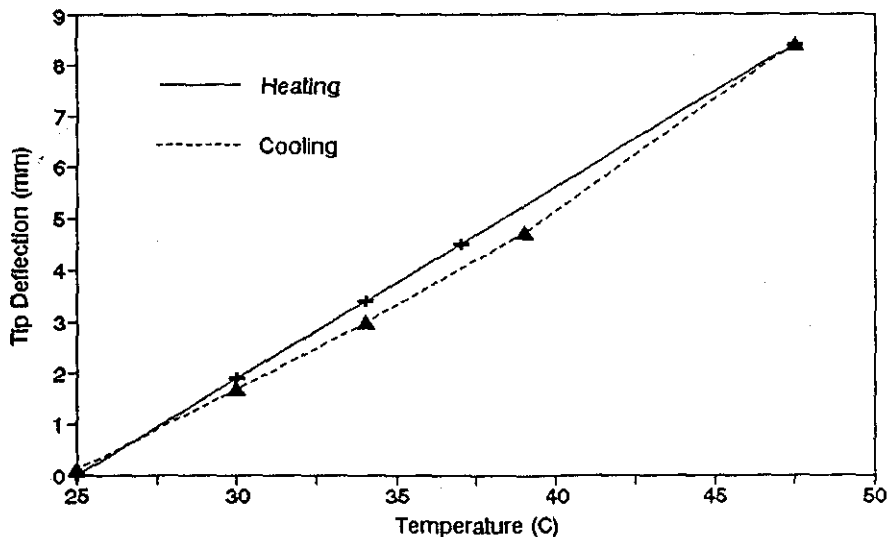


Figure 3. Deflection vs temperature in ambient humidity. Coating is 0.54 mil thick with 5 min seal.

Table 1. Effect of Storage Environment on Cracking

Time (wk)	Storage		Coating (thick- ness/seal time)	T <sub>c</sub> (°C)	cracks/3 coupons at T (°C)
	Temp. (°C)	Humidity (ppm)			
5	30	2 - 5	0.5mil/5min	130 - 140	0 (100°C) 29 (200°C)
5	amb.	amb.	0.5/5	< 100	40 (100°C) 72 (200°C)
5	30	90%RH	0.5/5	70 - 80	120 (100°C) 360 (200°C)
4	70	90	1/5	-	8 (70°C)
4	80	90	1/5	-	13 (80°C)

In Fig. 4 we plot each of the temperature-humidity conditions from Table 1 and show its effect on cracking tendency. It is likely that the process that causes easier cracking at elevated temperature and low humidity is different from that responsible for the degradation at moderate temperature and high humidity. However, as a guide for operations, it may be convenient to think of the interaction of storage temperature and humidity as shown schematically in Fig. 4.

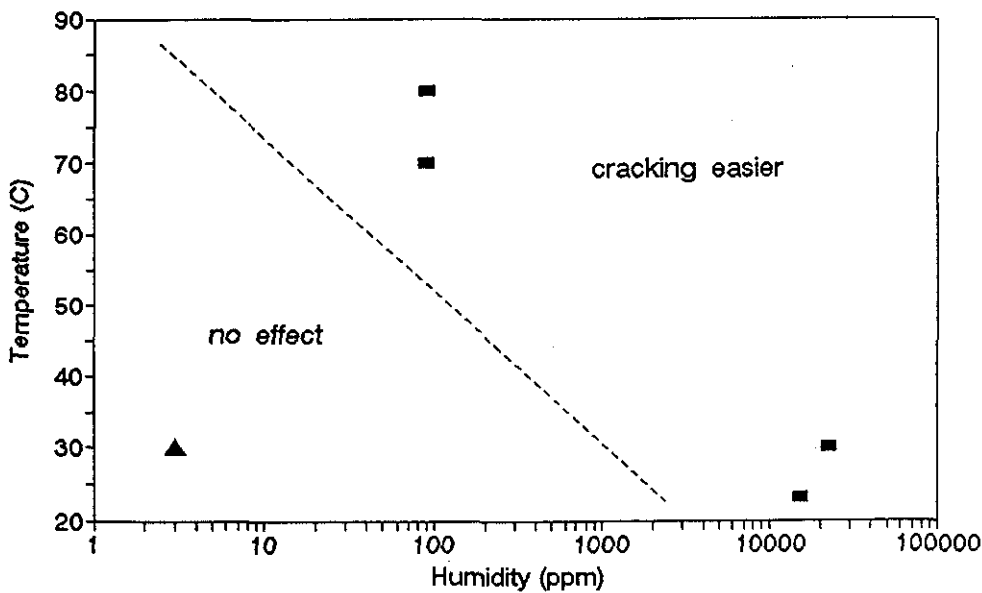


Figure 4. Effect of storage temperature and humidity on cracking: conditions producing enhanced cracking (■) or having no effect (▲).



### C. Cracking During Cyclic Heating.

Table 2 is a summary of cyclic test conditions and observations. In each test there was an incubation period before cracks appeared. The cycles at which cracks initiate,  $N_i$ , was different for each coating thickness but did not depend on seal time. The subsequent increase in crack density was proportional to  $\log(\text{cycles})$ . Final crack densities did not seem to depend upon coating thickness or seal time, so the end of test crack densities reported in Table 2 are the ranges for all coupon sets in the test.

During tests 4 and 5, sets of coupons were left in the hot chamber for the test duration. The 70° and 80°C results listed in Table 1 are the "static" coupons that accompanied test 4 in Table 2. The cycled coupons were at these temperatures for only half the time of the static samples, but they had up to 3 times the crack density. Thus, a cyclic fatigue process does occur in these oxide coatings that enhances cracking.

A detailed analysis of these tests is found in ref. 4. Here we point out qualitative features of the results. Comparing test 1 with tests 2-4, crack initiation is promoted at 70°C by a dry atmosphere. Tests 4 and 5 illustrate the strong dependence of cracking on  $T_{\max}$ . Comparing tests 3 and 4,  $T_{\min}$  has no effect on cracking. This is not surprising, since aluminum oxide is quite strong in compression.

Most of the cracks developed during these cyclic tests were small, about 5mm long and only 0.2  $\mu\text{m}$  wide, which distinguishes them from the longer and wider cracks obtained in the  $T_c$  tests. Only for the 1 mil/20 min coatings did some early cracks develop during cycling that extended across the specimen.

### D. Deformation of Metal Substrate

When using 10 mil sheet, it was observed that cracks that developed on one surface left a shallow indentation on the opposite surface. Examination of the metal after stripping the oxide revealed that severe plastic deformation had occurred, resulting in a groove in the metal along the crack length, and the indentation was due to necking of the sheet along the crack. Similar grooves developed in thicker sheet, but the deformation did not extend to the other surface. A finite element analysis showed that occurrence of a crack in the coating by thermal loading induced a very large tensile stress in the substrate under the crack, exceeding the nominal yield strength of the alloy (4). Substrate yielding is the origin of the groove.

Table 2. Thermal Cycle Tests  
0.5 and 1 mil coatings with 5 or 20 min seal

Test	$T_{\max}$ (°C)	$T_{\min}$ (°C)	Humidity (ppm)	Cycles		cracks per 3 coupons	
				total	$N_i$		
					0.5mil		1.0mil
1	70	25	ambient	2300	>2300	0	
2	70	25	175	1900	1600	1200	2 - 14
3	70	-40	50	4800	1000	1200	12 - 21
4	70	25	80	4300	1400	300	10 - 24
5	80	25	50	6100	300	<100	32 - 44

Grooves were found under all cracks that were examined, and were about the same width as the crack in the oxide. In addition, a zone of plastic deformation extended for a considerable distance on either side of the groove.

#### E. Metallurgical Factors

1. Temper - Metal temper affects crack width and density. Whole hard 20 mil 5657 sheet was annealed at several conditions and then anodized. Subsequent heating at 175°C for 20min produced the following crack characteristics:

<u>anneal</u>	<u>approx temper</u>	<u>cracks/coupon</u>	<u>crack width</u>
none	H19	4	2-5 $\mu\text{m}$
260°C/6 hr	H25	9	1
288°C/6 hr	H24 (est.)	15	<1

The inverse relation between width and density of cracks suggests that total energy release during cracking may be independent of temper, but the distribution of the energy release over the surface depends upon metal yield strength - the lower the yield strength, the more evenly distributed the release.

2. Alloy - Table 3 lists commercial alloys that have been examined during this study and their  $T_c$  values. Allowing for the approximate nature of  $T_c$ , there is still a real difference among alloys with regard to cracking tendency. The failure of brittle materials, such as oxides, is presumed to initiate at defects. For the anodic oxide coatings it was thought that the precipitate particles from the metal that are incorporated into the oxide would serve as the defects to trigger fracture. However, one of the lowest  $T_c$  values was obtained with 4N metal, which has no significant density of precipitate particles. Particles are found in the coating on 5657 alloy at a density of about  $2 \times 10^5 \text{ cm}^{-2}$  and are mostly 0.4-5  $\mu\text{m}$  in size, with a few particles as large as 25-100  $\mu\text{m}$ . With 1100 alloy, which has a higher density of particles, no cracks were produced up to 200°C. At this temperature the stress in 0.5 mil oxide is greater than 65 ksi. Further study of this puzzling behavior is in progress.

Table 3.  $T_c$  of Commercial Alloy Sheet  
0.5 mil/5 (and 20) min coating

<u>Alloy</u>	<u>Temper</u>	<u>Thickness</u>	<u>Test Sets</u>	<u><math>T_c</math></u>
		(mil)	(3 coupons per set)	(°C)
5657	H242	25	9	132
5657	H25	25	1	130
5657	H25	20	1	130
3002	H25	25	1	175
3002	H18	12	4	171
6061	T6	20	1	> 200
1100		10	1	> 200
1199	H19	20	4	115

#### IV. CONCLUSIONS

1. Coating stress is tensile after anodizing and compressive after sealing.

2. Coating stress responds to a large decrease in humidity by shifting from compressive (humid) to tensile (dry). This is the major determinant of coating stress in dry environment, rather than the coating process conditions.

3. The response of stress to humidity is reversible.

4. The response of stress to a temperature increase is different at low and high humidities. At low humidity, the slope  $\Delta\sigma/\Delta T$  is about half its value at ambient humidity.

5. During stepwise heating of coatings on 25 mil 5657 sheet, cracks initiate at a stress as low as 26 ksi for 1 mil oxide and 35 ksi for 0.5 mil oxide.

6. With some alloys, cracks did not occur at temperatures corresponding to stresses greater than 65 ksi ( $>200^\circ\text{C}$ ). Reasons for this high crack resistance are being sought.

7. Long term storage at ambient or higher humidity degrades the oxide so that it cracks more easily upon heating. Dry storage at moderate temperature prevents this degradation.

8. Anodized coatings exhibit cyclic fatigue under thermal cycling in dry atmosphere at relatively modest temperatures. Crack generation is dependent on  $T_{\text{max}}$  but not on  $T_{\text{min}}$ .

9. The formation of oxide cracks by heating is accompanied by plastic deformation of the substrate to produce a groove in the metal along the crack line.

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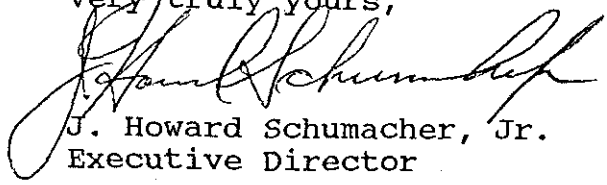
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