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UNIVERSITY OF LOUISVILLE

HIGH TEMPERATURE COATINGS FOR AIRCRAFT

A Thesis
Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

Arthur Hoff Isaacs
September 1948

HIGH TEMPERATURE COATINGS FOR AIRCRAFT

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Approved by the Examining Committee.

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September 1948

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ABSTRACT

This thesis is a presentation of formulations for heat resistant coatings which will withstand a temperature of 800° F. when applied to mild steel. The methods of preparation and testing of the formulations are given along with tabulations and discussion of results.

A satisfactory general heat resistant coating was obtained with a two coat system. The primer coat consisted of a carbonizing organic vehicle pigmented with a combination of aluminum, zinc, and cadmium. The top coat was a silicone-glyceryl phthalate vehicle pigmented with aluminum.

Best results for a glossy sea blue heat resistant coating were obtained also with a two coat system. The primer coat was the same as given above for the general heat resistant coating. The top coat for the blue coating was formulated from a silicone-glyceryl phthalate vehicle pigmented with green, blue, and black refractory oxides in fixed ratios.

INTRODUCTION

Organic protective coatings have consistently demonstrated an important role in engineering. These coatings have been primarily a development of the arts. Today, however, their production is finally emerging as an industrial science.

The five main purposes of protective coatings are decoration, protection of structural and technical materials, sanitation, obtaining better illumination of objects, and producing a desired surface condition.

The protection of structural materials and the obtaining of desired surface conditions are of primary interest to the Navy Department, Bureau of Aeronautics. In the case of protection, it has been estimated (1) that up to 2 percent of the world's tonnage of iron must be replaced yearly because of corrosion. Latest estimates indicate these losses are greater than a billion dollars annually. As for desired surface conditions, Lt. Com. A. M. Malloy states (2), "The application of the best possible smooth finish at the time of manufacture will enhance the maximum performance of a 400 m.p.h. fighter airplane to the extent of at least 13 m.p.h. This increased performance is the equivalent of 140 h.p."

Thus it is recognized that the advances of modern technology in the design and construction of naval aircraft and component parts would be nullified greatly if deleterious effects of the elements and severe operating conditions encountered in service were not offset by protective coatings. As has been stated (3), "The small margin dividing victory and defeat in (aerial) combat may often be determined by the thin film of protection which is afforded by protective coatings."

The development of a protective coating which will withstand the severe conditions encountered by naval aircraft poses a problem. Engine exhaust manifolds and jet assemblies with adjacent fuselage parts are not only subjected to intense heat but also to low temperatures encountered at high altitudes and in arctic regions. Further, naval aircraft are frequently in contact with salt spray which is extremely corrosive to ferrous metals. Structural strength cannot be sacrificed for corrosion resistance. It is therefore necessary in many instances to rely on protective coatings, rather than corrosion resistant metals, for adequate protection.

There were no coatings which would withstand the conditions mentioned above at the time jet propelled aircraft were introduced to modern aviation. For the development of such coatings the Bureau of Aeronautics entered into a contract with the Institute of Industrial Research of the University of Louisville for development of a protective coating satisfactory for this application. The requirements for the coating were as follows:

1. It shall withstand exposure to a temperature of 800° F. for a period of several hundred hours.
2. It shall eliminate corrosion of the base metal.
3. It shall adhere when exposed to the vibrations encountered in aircraft service.

4. It shall pass a bend test over a $\frac{1}{8}$ in. mandrel after subjection to 800° F.
5. It shall possess conventional application characteristics and shall result in a smooth, thin continuous film.
6. It shall not require a curing temperature greater than 300° F.

After the project had progressed for some months the additional requirement, that it be sea blue in color, was added.

HISTORICAL

The use of protective coatings dates back several thousand years. Though some progress was made through the ages, the development of the protective coating industry was quite slow.

With the advent of the twentieth century came the automobile, aircraft, and a tremendous expansion of industry. As a result of this expansion progress was also made in the field of protective coatings. However, at the time this research was started there were no protective coatings which were entirely satisfactory above 600° F.

A number of patents have been granted for heat resistant coating formulations in the past fifty years. One of the first was given to Princesse Demidoff (4) for a formulation using magnesium carbonate as a base. This coating was said to inhibit the corrosion of metallic surfaces at elevated temperatures.

Other investigators used a variety of pigments and vehicles. These materials included as pigments, carborundum powder, various metals and alloys of metals, camphor, talc, and titanium. Linseed oil, fish oil, coal tar, Japanese varnish, and alkali silicates were employed as vehicles.

Coatings using alkali silicate vehicles are rather numerous and are basically the same. The minor differences which exist are in pigmentation, variation of pigment vehicle-ratio, and addition of materials to promote setting.

A survey in 1945 indicated that none of the heat resistant coatings available were satisfactory for use above temperatures of 600° F. The modern mode of warfare had greatly increased the need for a satisfactory heat resistant coating of this type. The Houston Paint and Varnish Club began at this time an investigation of heat resistant finishes (5). The investigation showed that films composed of organic binders disintegrated to an extent that rendered them valueless between 600° F. and 800° F. for prolonged periods of heating. In its latest report (6) this club concludes that DC 801* silicone heavily pigmented with aluminum, will produce a satisfactory coating if applied in a thickness of 2-3 mills.

Work which has already been completed on this research program (7, 8) has produced coatings which will withstand a temperature of 800° F. and are reasonably satisfactory with respect to adhesion, weathering, and flexibility. Satisfactory vehicles included (1) a carbonizing phenolic varnish, (2) a silicone resin, and (3) a mixture of glyceryl phthalate spar varnish and a silicone resin. Various metallic powders were satisfactory as pigments.

* See appendix page 58

THEORETICAL

Paints and related finishes protect metals by the interposition of a continuous inert and adherent film between the material surface and its environment. In the case of metallic finishes over metals, electrochemical relationships between environment, coating, and base metal play a predominant part in determining coating efficiency. These electrochemical relationships, in general, play a relatively minor part in the field of organic finishes. The degree to which the environment can be blocked off mechanically from reaction with the corrodible metal assumes major importance in the organic coating field.

Organic finishing materials consist essentially of a continuous, inert, film-forming phase in which is dispersed a pigment or combination of pigments. The liquid phase is essential for application purposes. Just as necessary is the development of an immobilized structure. The conversion from liquid to solid phase is therefore an absolute requirement.

Organic finishes may be grouped into three general classes - paints, enamels, and lacquers.

PAINTS

A paint essentially is a dispersion of pigments in a drying oil. The drying oil is converted to a gel state, usually by oxidation at normal temperatures. A drier may be added to accelerate the early stage of drying. The oil may be partially polymerized or bodied, or may be fortified by addition of small percentages of soluble resins. These resins are usually synthetic, and are added to increase film strength and to hasten gelation. Drying, as a rule is relatively slow; and films, when fresh, are soft and readily deformable. With age, progressive hardening occurs attended by a gradual, definite reduction in distensibility.

Paints are usually manufactured by "grinding" pigments into oils using differential speed rolls. For certain hard abrasive pigments, buhrstone mills may be employed. In many instances ball or pebble mills are used. Actually the pigment particles are not "ground", but clusters of them are separated to permit intimate wetting of all particles by the liquid phase.

Gloss of paints, as a rule, is limited, and is rapidly lost on outdoor exposure.

ENAMELS

An enamel consists of an intimate dispersion of pigments in a varnish or resin vehicle. Other than the higher degree of dispersion of pigment in the vehicle, the essential difference between a paint and an enamel lies in the nature of the vehicle. The enamel vehicle may be an oil-resin mixture, containing either natural or synthetic resins, or possibly a mixture of the two. The oleoresinous vehicles are commonly known as varnishes. These varnishes, containing drying oils, are converted largely by oxygen absorption. Newer synthetic resin vehicles may be either oxygen convertible, heat convertible, or both, depending upon the particular resin. Enamels, as a class, dry more rapidly than paints and are appreciably harder and tougher. In certain compositions the use of heat accelerates gelation. At the same time toughness and hardness are increased to a point where a vitreous character is approached. Great advances have been made with this type of coating in the field of organic finishes.

Because of finer pigment dispersion and the innate gloss of the dry vehicle, enamels satisfy decorative as well as protective demands. Their resistance to mechanical abuse has made them highly applicable to metal finishing.

In oleoresinous varnishes, the ratio of drying oil to resin is of prime importance. In general, with natural and many synthetic resins, the higher the oil content, the more flexible and durable the varnish. Conversely, air-drying speed, initial hardness, and brittleness increase with resin content. Certain of the more recently developed varnishes, however, do not follow this rule.

LACQUER ENAMELS

A lacquer enamel consists essentially of a dispersion of pigment in a lacquer vehicle. This vehicle is distinctly different from the oleoresinous or oil media. The differences lie in composition, formulation, and film forming mechanism. The non-volatile residual film ordinarily contains three components. These are the cellulose ester, usually nitrocellulose, the resin, and the plasticizer. The cellulose derivative lends strength by virtue of its strong and relatively long resilient molecules. The resin provides adhesion and hardness. It also permits the use of thicker films than would be possible using a cellulose derivative alone. The plasticizer contributes distensibility to the film structure.

Because of their great drying speed, lacquer enamels find wide application and decoration of almost any articles where spray application is feasible.

ORGANIC FINISH COMPONENTS

Organic finishes, in the broadest classification, consist of two components: an organic vehicle and a pigment or pigments. The vehicle constitutes the continuous phase, and the pigment the discontinuous phase. The vehicle must be convertible from a mobile state to an immobile solid state of a film. The fluid state is necessary for purpose of application of the finishing material. Of similar importance is the conversion to a solid form, namely a film.

The resinous state must be developed before a film can be formed. This state may be preconditioned in the fluid vehicle by the presence of resin molecules dispersed in a solvent. The same state may be developed in the film after application through polymerization. Such growth of resinous molecules may occur by chemical reaction with components in the environment, or by heating. The primary characteristic of any resin is that it consist of very large molecules. These molecules may be long straight chains, branched chains, or cross-linked chains. The opportunities for polymerization as the molecule grows larger are increased tremendously. Large size molecules obviously possess a large molecular surface area which gives molecular surface forces the opportunity for action. Van der Waal's forces at these surfaces can produce solvation, association, and aggregation effects. Because of these effects, molecules may associate into micellular structure or may adsorb foreign molecules with great tenacity. The three conditions - large molecules, complexity of structure, and large surface area - characterize the resinous state which determines solid film formation. The physical behavior of resins (9, 10) is primarily determined by the size and geometric structure of the molecules, and only secondarily by chemical composition.

Driers, usually organic compounds of lead, manganese, and cobalt, have been used in conjunction with drying oils and oleoresinous varnishes for many years (11). Their function is primarily catalytic. They accelerate the addition of oxygen at the double bonds of unsaturated drying oil acids. Only small percentages of driers are necessary. The character of the film in terms of its physical properties, such as tensile strength and elongation, is affected by the kind and amount of drier used. Cobalt driers affect particularly the surface drying properties while manganese and lead compounds influence the through drying characteristics (12).

Unpigmented organic coatings find only limited use as protective coatings for metals. The addition of pigments introduces a number of highly important changes in these clear coatings i.e. coloration and capacity for obscuring the underlying base materials. By pigment addition the organic coating is strengthened, hardened, and made relatively more durable. The deleterious effects of certain environmental factors are delayed by pigmentation. For example, the rate of moisture penetration is reduced, and in some instances the destructive action of ultraviolet light is minimized. By incorporation of suitable pigments in the priming coats of multiple coat systems, the corrosion of the base metal may be retarded.

One of the outstanding characteristics of almost all pigments is their fineness of particle size. The majority of pigments fall in a range of 0.1 to 5.0 microns in average diameter. A pigment should not be used unless it will pass a standard 325 mesh screen (13). Fine particles provide a large surface area. With such large surface areas, interfacial properties of the pigment-vehicle surface become extremely important. The wettability of the pigment by the binding vehicle is determined by the physical and chemical character of the pigment surface. These wetting characteristics affect not only ease and stability of dispersion, but also the type of failure occurring upon exposure to the environment. Pigments of alkaline character are wetted readily and for this reason materially aid the physical structure of the paint film. Other pigments are difficult to wet completely. This effect is reflected to a considerble extent by chalking tendencies of coatings containing such poorly wet pigments.

It has been found that pigment-vehicle ratios are important. With regard to durability, an optimum value for pigment concentration has been established. For a large number of pigments this value lies between twenty-five and thirty-five percent by volume of pigment (14). This concentration is approximately the optimum range of application consistency. Hence a useful relation between durability and pigment content is available at a convenient point in formulating new coatings. In priming coats, when inhibitive pigments (chromates or red lead) are used, the added electrochemical protection offered by these pigments permits wider latitude in pigment content variation.

There is no single pigment that serves all purposes. Reactive pigments may be used for priming protection or for hardening and strengthening of the film. Non-reactive pigments may be used for coloration, for easing stresses in the coating, or for retention of coating elasticity. Other pigments have the particular virtue of excluding ultraviolet light or of reducing moisture penetration. It has been learned (15) that the admixture of several pigments, particularly in top coats, is frequently accompanied by a "symbiotic-like" effect, in which the several pigments exert a mutual benefit to improve the entire coating.

COATING PROPERTIES

The durability of a protective coating involves numerous factors. Those of major importance are adhesion, flexibility, abrasion resistance, cohesion, and hardness. Conversely, there are certain characteristics to be avoided. Some of these undesirable attributes are checking, chalking, blistering, and fading.

One of the criteria for a suitable coating material is the strength of its bond to the base on which it is applied. This bonding is known as adhesion. Adhesion may be of two types. These types are mechanical adhesion and specific adhesion.

The penetration of the coating film into the interstices of the substrate, resulting in a physical interlocking of the coating and the substrate, is known as mechanical adhesion. There are two factors which can greatly affect this type adhesion. The first of these is the interfacial area between the coating and the substrate. A roughening of the metal surface can increase the contact area at least twenty-fold (16). Another factor which affects mechanical adhesion is the fluidity of the film forming medium. A fluid medium can penetrate the substrate to a greater degree than a viscous medium. Fluidity may be increased by the use of heat and/or solvents. Care must be taken in the use of solvents, as shrinking stresses developed on their evaporation may cause rupture of the bonding.

Cleanliness of the base surface is an extremely important factor in the adhesion of coatings. While a contaminated layer may itself be readily wetted by a coating vehicle, the adhesion of such a layer to the base material is frequently of low order. Hence the adhesion of the film to the base is low. Recent experiments (17) have indicated that contamination by grease or oil to the extent of tenths of a milligram per square inch of surface definitely affects the adherence of most organic finishes.

Specific adhesion is that adhesion which depends on the electrochemical relationship of the coating and the base metal. The electrical forces residing in the atoms and the molecules of the metal interact with the electrical forces residing in the polar molecules of the finishing medium. The distance over which these attracting forces can act is extremely short. This distance, which amounts to several molecular layers, would be approximately one-thousandth of the thickness of an average single-coat paint film. Therefore, the primary requirement is that the coating medium come into direct contact with the base metal.

An essential characteristic of a protective coating is its flexibility. However, the fact that a coating has good flexibility should not be taken as the sole criterion that it will be satisfactory. Although a coating which exhibits good adhesion will in all probability be flexible, the converse need not hold (18). The flexibility of a coating is primarily determined by the vehicle employed.

The characteristics of hardness and abrasion resistance are closely interlocked with adhesion, cohesion, and flexibility. In general, as hardness increases, flexibility will decrease. Further, a softer coating is, as a rule, more abrasion resistant than a hard one. It is therefore necessary to compromise on some properties to obtain a coating with the optimum characteristics.

Film thickness is a vital factor in coating behavior. Finishes must be tested at controlled film thicknesses. For good correlation, this thickness should correspond to that used in service. Krumbhaar (19) emphasizes the fact as follows: "All data on mechanical film properties (and indirectly on the performance and durability) are dependent on the thickness of the film. The same finishing material at two different coating thicknesses may exhibit widely different behavior; the durability may vary and the type of failure may be completely changed."

The selection of a coating which will offer resistance to water is a fundamental requisite in the design of coatings for protection of steel surfaces. This resistance is a function of both vehicle and pigment. Leafing pigments offer great resistance to water penetration (20). Probably the best known examples of leafing pigments are aluminum powder and waterground mica. Vehicles employed with such pigments should be sufficiently fluid to permit mobility of the pigment particles. This mobility will permit the leafing characteristic of the pigment to exert itself.

There is also cathodic protection against corrosion. Examples of materials which provide this protection are zinc dust and red lead. This action is thought to be due to the neutralizing effect obtained by the use of such pigments (21).

It is known that zinc dust is anodic (in character) to iron. Furthermore, zinc metal has the property of diffusing intergranularly into the metallic iron surface at high temperatures (22).

In the preparation of a heat resistant coating, the problem is more specific. The number of materials which may be used is definitely limited because of oxidation and/or decomposition at the required temperature of 800° F. There are numerous metals suitable for use as pigments at high temperature. Zinc and aluminum have already been found adaptable to this problem (7, 8). Such inerts as mica and asbestine are also suitable.

The problem then resolves itself into utilizing these pigments in the proper vehicle.

Polytetraflourethane is probably the most heat resistant resin known at the present time. At 620° F. it undergoes a solid-phase transition with a sharp drop in dielectric strength. Dielectric strength is an important factor. Drops in dielectric strength indicate molecular decomposition which eventually results in film failure. In the neighborhood of 750° F., it decomposes to yield gaseous products. Small amounts of flourine-containing gases are given off at temperatures as low as 400° F. This material was not studied in the present work, for it is difficultly soluble, and has very poor adhesive characteristics (23).

The silicones are quite applicable to high temperature work. There are methyl silicone resins which suffer no decomposition or disintegration over a period of years at 400° F. These resins maintain their dielectric strength for long periods at temperatures as high as 550° F. Above 500° F. the methyl silicones oxidize, leaving a residue of silica. Phenyl silicone has greater thermal stability, but is not so ductile as methyl silicone. A copolymer of phenyl and methyl silicone may be prepared which has the desirable characteristics of both the phenyl and methyl silicones. These copolymers have as their upper service limit a temperature of approximately 500° F. However, in the absence of oxygen, these and other silicones are thermally stable at temperatures as high as 900° F. (24, 25). The possibility exists that a suitable antioxidizing or plasticizing material may be found to prevent or retard this type of failure.

The coumarone-indene resins are also stable at high temperatures in the absence of air; but, like the silicones, they oxidize in its presence.

Another possibility for a heat resistant coating lies in the use of a vehicle which will carbonize and leave the pigment "fritted" or fused to the surface of the metal.

A logical approach to the problem lies in the further investigation of the most satisfactory formulations found by Lutz (7), and Hayne (8). A base coat utilizing a zinc, aluminum, and cadmium pigmentation of a heat reactive varnish offers good possibilities. A finish coat which is an aluminum pigmented silicone-glyceryl phthalate formulation also appears to be satisfactory.

Glyceryl phthalate is one of the more heat resistant resins. It is not, however, as heat resistant as the silicones. Attributes for which it is known are flexibility and air-drying. Further, it is more fluid than the silicones. It was therefore thought that a copolymer might be formed with the silicone and the glyceryl phthalate. This copolymer would embody not only the heat resistance of the silicones, but also the flexibility and air drying characteristics of the glyceryl phthalate.

EXPERIMENTAL

Considering the material presented in the previous section, and the recommendations of Lutz (7), and Hayne (8), the following plan was set forth:

1. Extensive tests would be run on Hayne's two coat system; an aluminum pigmented silicone-glyceryl phthalate vehicle used as a finish coat, and an aluminum-zinc-cadmium pigmented carbonizing phenolic vehicle used as a base coat. These tests would determine exactly the characteristics of the system.
2. Modifications of this system would be tested in an effort to improve its properties.
3. Work would be done to give the best two coat system a sea blue pigmentation.

Before presenting the experimental data, the procedure and apparatus will be discussed.

The pigments to be incorporated in a formulation were weighed into a one-half pint can. Enough vehicle next was weighed into the can, so that on proper mixing a stiff paste would be formed. These weighings, were made on a triple beam balance accurate to 0.01 gram. A half-dozen steel balls, approximately one-quarter inch in diameter, were placed in the can. The can was sealed and placed on a rotating mixer (26). This apparatus simulated the action of a pebble mill, and "ground" the pigments into the vehicle. The mixing period was varied to meet requirements of the different formulations. Periodic microscope checks were made to determine whether satisfactory grinding and dispersion were being obtained. When the pigment was properly dispersed, the remainder of the vehicle was added and the cans were again placed on the mixer. After another one to two hours mixing, formulations were ready for application.

With the exception of certain special cases, the test panels were 3 in. by 3 in. mild steel panels. Panel thickness was approximately 1/32 in. These panels were polished with emery cloth, and solvent cleaned with acetone to give a clean, uniform surface for coating evaluation. The coating was applied using a conventional spraying hood and gun. Following the coating application, the panels were air dried for sixteen hours. Then a more intensive curing period was applied to the coating. This curing consisted of a three hour period under an infra-red lamp (27). Panel temperature for the duration of the curing period was approximately 275° F.

If the panels were not to be given a finish coat they were placed in the muffle furnace. This furnace (28) was Micromax controlled to ± 10° F. In most cases, the temperature used for testing was 800° F. The testing period was sixteen hours. After being heated, the panels were given a visual inspection. Those coatings passing the heating test were subjected to a "shock" test. This test consisted of quenching the panels from 800° F. into water at room temperature. Coatings that did not fail quenching were subjected to a bend test which was performed over a tapered mandrel (29). The diameter of this mandrel tapered from 1½ in. to ¼ in. in eight inches length. After the bending test, the panels were given a scratch test to check adhesion. Only the outside bend is considered in this test.

Coats which were successful to this point were tested in an accelerated weathering unit, Figure 1. This unit consisted of a rotating basket inside a copper drum. The panels were attached to the basket, which made one revolution every two hours. During basket rotation, the panels passed directly under the spray of nozzles. In addition, the chamber was completely filled with water mist.

A salt spray chamber, Figure 2, was used for more severe weather testing. This unit consists of a rectangular tank, with one bottom portion containing a twenty percent salt solution. Panels were hung in the upper portion. The tank was filled with a dense salt mist by means of a compressed-air operated spray-nozzle system. Water saturated air was supplied at 10 p.s.i.g. to the spray nozzle.

If a coating warranted further testing, a pipe stand (30) was used. This stand consisted of a fourteen inch length of 1½ in. standard steel pipe. The preparation of the pipe for coating evaluation was similar to that of the panels. After being cured the pipe was mounted in a vertical position on a ring stand. Wells were drilled into the pipe at various points, so that temperatures could be measured, at intervals, using a thermocouple potentiometer combination. Heating was accomplished with a Meker burner. A length of one inch copper tubing was mounted concentrically inside the steel pipe. This tubing

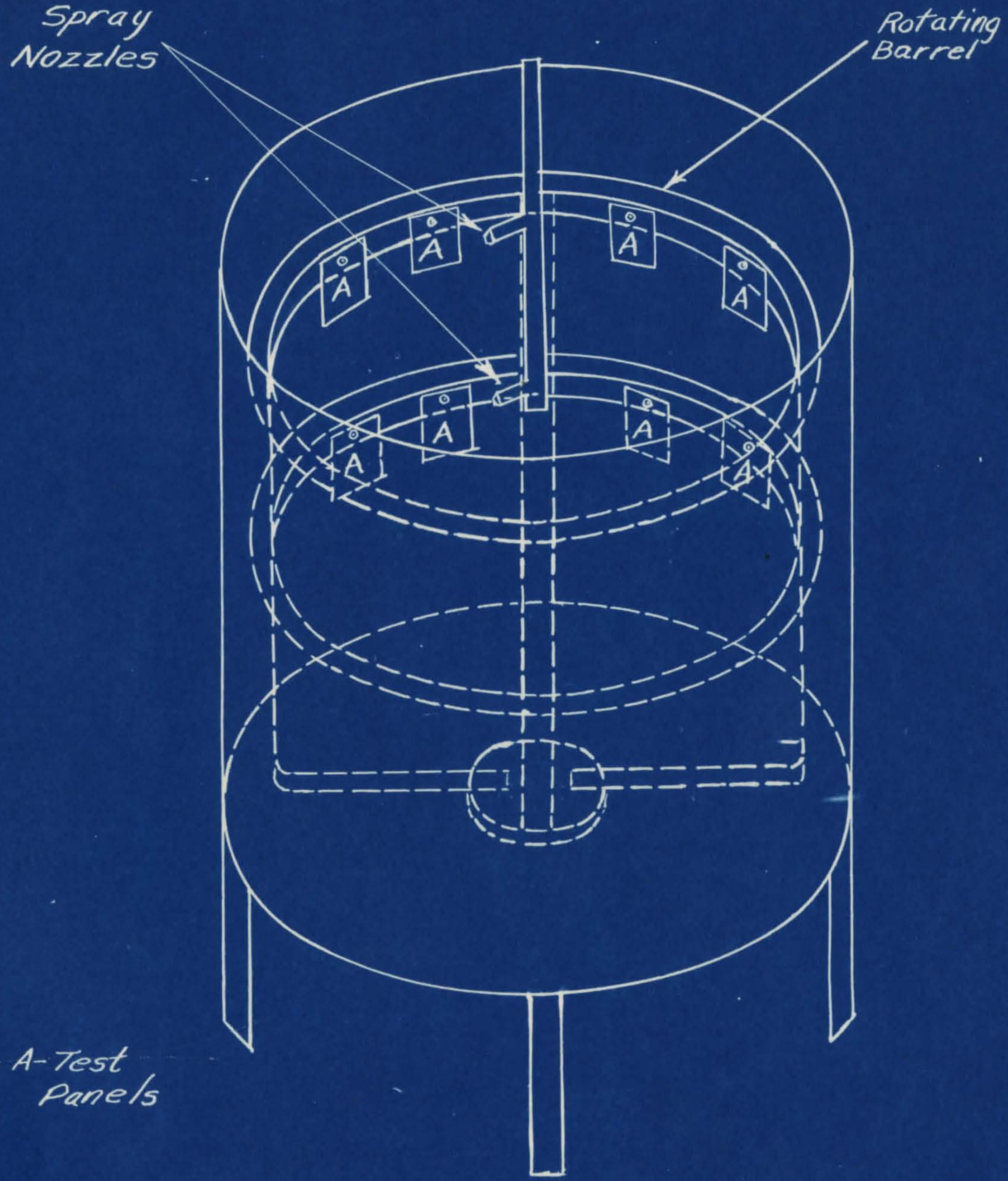

FIGURE 1 ACCELERATED WEATHERING UNIT

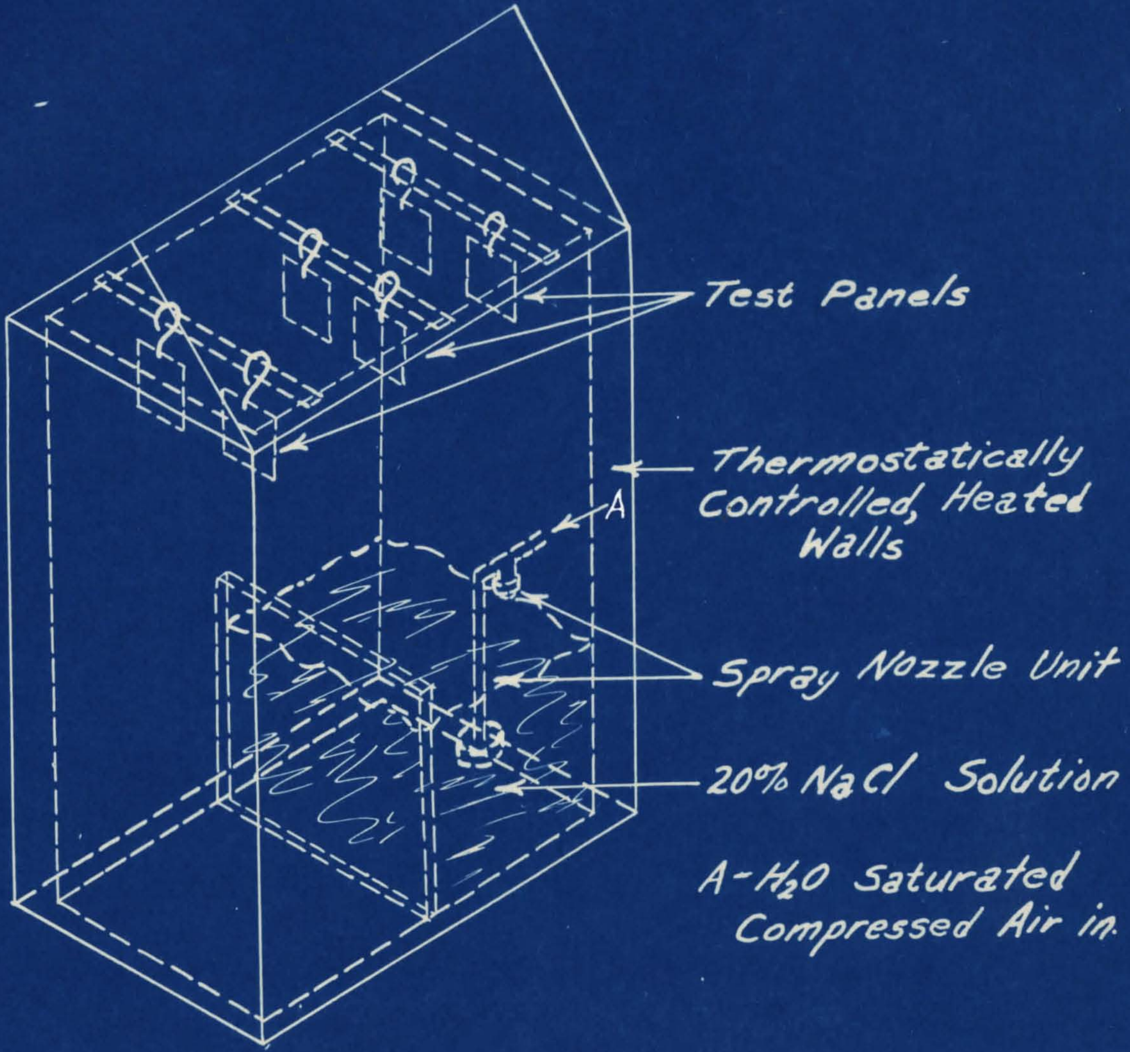

FIGURE 2 SALT SPRAY CHAMBER

permitted heating of the pipe with a reasonable temperature gradient between its two ends. Using this method of heat transfer, it was possible to obtain temperatures of approximately 800° F. at the longitudinal center of the pipe. Water was sprayed on the pipe at frequent intervals. This test more nearly approached actual service conditions than any other. When panels are heated in a muffle furnace, the panel and coating are in a bath of uniform temperature. When the panel is quenched in cold water, the entire panel again goes into a bath of uniform temperature. In the case of the pipe stand, however, the inner surface of the coating is exposed to radiation from a piece of metal which is at a very high temperature. The outer surface of the coating is exposed to room temperature. The same condition exists when the pipe is sprayed with water.

TWO COAT SYSTEM

Following the experimental plan set forth, a group of panels coated with the system H21, H1 was prepared. The composition of these formulations is shown in the following table.

TABLE I - COMPOSITION OF BASIC TWO COAT SYSTEM

Sample Number	Type of Coating	Pigment			Vehicle	
H21	Base	1 Al	5 Zn	2 Cd	30 Phenolic	
H1	Finish		6 Al		10 DC802	15 G.P.*

Note: Al - Aluminum paste
Zn - Zinc powder
Cd - Cadmium powder
Phenolic - Heat reactive phenolic varnish
DC802 - Dow Corning 802 silicone resin
G.P. - Glyceryl phthalate spar varnish
All quantities are parts by weight

After testing several groups of panels coated with this system, it was observed that some panels failed slightly on quench or bend tests. Furthermore, on standing overnight a can of coating H1 would gel.

The addition of small amounts of micronized mica to coating formulations reportedly improves bending and adhesive characteristics (31). Hayne (8) noticed that the presence of zinc

---

* All abbreviations are explained in the appendix.

in formulations prevented skinning in the can. With these facts in mind, formulations were tested using various top and base coats. The following tables list the composition of these formulations.

TABLE II - BASE COATS CONTAINING METALLIC PIGMENTS, MICA AND ASBESTINE

Sample Number			Pigment		Vehicle
H19	1 Al	5 Zn			30 Phenolic
H21	1 Al	5 Zn	2 Cd		30 Phenolic
H51	1 Al	5 Zn	2 Cd	0.9 Asb.	30 Phenolic
H52	1 Al	5 Zn	2 Cd	0.9 M.M.	30 Phenolic

Note: Asb. - Asbestine

M.M. - Micronized mica

All quantities are parts by weight

TABLE III - FINISH COATS CONTAINING METALLIC PIGMENTS, MICA AND ASBESTINE

Sample Number	Pigment			Vehicle	
H1	6 Al			10 DC802	15 G.P.
H36	5 Al	0.5 Zn		10 DC802	15 G.P.
H38	5 Al	1.0 Zn	0.5 M.M.	10 DC802	15 G.P.
H50	5 Al	1.0 Zn		10 DC802	15 G.P.
H54	5 Al	1.0 Zn	0.5 Asb.	10 DC802	15 G.P.
H56	6 Al		0.5 Asb.	10 DC802	15 G.P.
H57	5 Al	1.0 Cd		10 DC802	15 G.P.

Note: All quantities are parts by weight

These particular formulations were chosen because Hayne (8) reported them to be satisfactory as single coat systems except for weathering. It was felt that if the proper match of base and top coat were obtained, good weathering also could be achieved. Results using these formulations as two coat systems are set forth in a table on the following page.

H21 was the only undercoat giving a good bend test with various top coats. Moreover, it was the only undercoat with no quenching failures. The indications were, therefore, that H21 was the best undercoat. Because of the failures of H51 and H52, the use of micronized mica and asbestine in the undercoat is definitely eliminated. These materials did not improve adhesion and flexibility of the coatings.

TABLE IV - RESULTS OBTAINED USING VARIOUS TWO COAT SYSTEMS

Failure on Quench		Poor or Failed on Bend		Fair on Bend		Good on Bend	
Coating Composition		Coating Composition		Coating Composition		Coating Composition	
1st	2nd	1st	2nd	1st	2nd	1st	2nd
H51	H36	H19	H1	H21	H54	H21	H1
H52	H36	H19	H50			H21	H50
H52	H38	H21	H36				
H52	H54	H21	H38				
		H51	H1				
		H51	H38				
		H51	H54				
		H52	H1				

Several panels of the system H21-H1, and H21-H50 were prepared and extensively tested by repeated heatings and quenchings. After a number of quenchings, the panels with H50 top coat began to flake slightly. Observations showed, however, that cans of formulation H50 did not skin, when permitted to stand as long as ten days. It was thus felt, that a coating could be formulated which would contain less zinc than H50 and still retain its anti-skinning properties. Consequently formulations H59 and H60 were prepared. These formulations contained only one-tenth gram of zinc. Panels coated with these formulations failed on quenching, thus eliminating the possible use of zinc in a top coat formulation.

With other formulations completely eliminated, extensive tests were begun on the H21-H1 system to determine its exact characteristics. One of the first tests was that on the pipe stand. The pipe was heated for a period of seven to ten hours. After repeated sprayings with cold water while the pipe was at elevated temperature, the pipe was then allowed to cool. An inspection showed that the only failures occurred where the pipe had been definitely above 900° F.

The question of resistance to hot oil and solvents arose. To test this resistance a group of panels were prepared. After they were cured, the panels were heated in the muffle furnace for fifty-four hours. The heating period included quenching at eighteen hour intervals. The panels were then heated in S.A.E. 30 motor oil at 200° F. for five hours. This test was followed by a ten hour heating period in oil at 300° F. Oil heating had no effect on the coating. The same panels were heated in toluene at 200° F. for five hours, and then placed in the accelerated weathering unit. After 120 hours exposure the panels were removed and inspected. Inspection showed that the coating weathered similarly to panels which had received no oil and solvent treatment. Slight washing was evident but no rust.

As a final weathering test, a group of panels were prepared using combinations of top coat and base coat. In addition formulation H49 was also tested. This coating had the pigmentation of H21 in the vehicle of H1. These panels were heated for 100 hours at 800° F. before being tested in the spray chamber.

TABLE V - RESULTS OF 200 HOUR SALT SPRAY TEST

Coating Composition 1st	2nd	Results
H1	---	Rusted badly
H49	---	Rusted badly
H21	---	Rusted
H21	H21	Slightly rusted - Badly washed
H21	H49	Very slightly rusted - Badly washed
H21	H1	No rust - Some washing

The data of Table V show that the system H21 - H1 has better weathering characteristics than the other systems. System H21 - H49 was also a silicone-glyceryl phthalate finish coat applied over a phenolic base coat. However, panels coated with this formulation showed signs of rust. This test shows that leafing of aluminum in the top coat definitely inhibits passage of water.

BLUE HEAT RESISTANT COATING

A logical starting place in developing a blue heat resistant coating was felt to lie in pigmenting the top coat of the previously developed system. The blue pigment chosen was iron blue. This pigment was selected because it has excellent baking characteristics and permanency of tint. Using this pigment, coating H58 (see Table VI) was formulated. This coating consisted of the existing top coat with addition of iron blue pigment. The effect of iron blue on the color of this formulation was not noticeable on the panels. From this fact it was evident that the proportion of aluminum in the top coat would have to be greatly reduced to permit blue coloration. On the basis of this test the remainder of the formulations of Table VI was prepared.

TABLE VI - PRELIMINARY BLUE COATINGS

Sample Number	Pigment				Vehicle	
H58	1 I.B.	6 Al			10 DC802	15 G.P.
H62	1 U.B.	1 Al	5 Zn	2 Cd	10 DC802	15 G.P.
H63	1 I.B.	1 Al	5 Zn	2 Cd	10 DC802	15 G.P.
H64	1 U.B.				10 DC802	15 G.P.
H65	1 U.B.		5 Zn		10 DC802	15 G.P.
H66	1 U.B.	0.5 Al			10 DC802	15 G.P.
H67	1 U.B.	0.5 Al	5 Zn		10 DC802	15 G.P.
H68	2 U.B.	1 Al	5 Zn	2 Cd	10 DC802	15 G.P.

Note: I.B. - Iron blue

U.B. - Ultramarine blue

All quantities are parts by weight

Formulation H49, previously discussed, was the best non-blue finish coat of low aluminum content. It was felt that another blue pigment might have better tinting strength than iron blue. Referring to the table on pigments (32) it was found that ultramarine blue had good permanency of tint and excellent baking characteristics. Formulations H62 and H63, blue variations of H49, were prepared to compare iron blue with ultramarine blue. After being sprayed, but before being fired, panels coated with the iron blue formulation had a deep bluish hue. Those coated with the ultramarine blue formulation were a light blue. After being fired for seventeen hours at 800° F., the panels coated with H63 were a dull grayish-tan color. Those coated with H62 retained the original light blue color. The panels of H62 were returned to the furnace. After eighty-three hours of heating, the presence of ultramarine blue was still evident although the panels were lighter in color. The grayish tan color of panels coated with H63 was apparently due to decomposition of the iron blue pigment.

To increase the effect of ultramarine blue on coating color, formulations H64 through H68 were prepared. In these formulations the ratio of ultramarine blue to aluminum was increased. All panels failed with the exception of those coated with H66. Panels coated with H66 withstood heating, quench, and bend tests. The blue color, while much lighter in shade, did remain after the panels had been heated forty-two hours at 800° F. At this point the only blue coatings which had held up were those using the metallic pigmentation of H49.

Formulations using Chinese blue and phthalocyanine blue, H69 through H72, were prepared in an attempt to find a blue pigment with greater tinting strength. Chinese blue is a member of the iron blue family. Phthalocyanine blues are reportedly stable at very high temperatures (33). Both of these pigments decomposed, however, when coatings containing them were heated to 800° F.

It was noted that, in order to intensify the blue color, the ratio of ultramarine blue to other pigments, and to vehicle would have to be increased a large amount. Therefore, the formulations of Table VII were prepared.

TABLE VII - FORMULATIONS CONTAINING HIGH ULTRAMARINE-VEHICLE RATIOS

Sample Number	Pigment			Vehicle	
H73	5 U.B.	1 Al		30 Phenolic	
H74	5 U.B.	1 Al	5 Zn	30 Phenolic	
H75	5 U.B.	1 Al		10 DC802	15 G.P.
H76	5 U.B.	1 Al	5 Zn	10 DC802	15 G.P.
I3	10 U.B.	1 Al	5 Zn	10 DC802	15 G.P.
I5	5 U.B.	1 Al	2.5 Zn	10 DC802	15 G.P.
I7	10 U.B.	1 Al	5 Fe_2O_3	10 DC802	15 G.P.
I8	10 U.B.	1 Al	5 Cd	10 DC802	15 G.P.

Note: All quantities are parts by weight.

Panels were coated with the formulations of Table VII and heated at 800° F. Observation showed that a dampened cloth would wash the blue pigment from the panels coated with H73 and H74. This poor adhesion was due to the fact that the vehicle used was of the carbonizing type and ultramarine blue will not "frit" to a metallic iron surface. The only coating which blistered or flaked on heating and/or quenching was I7. This coating failed on quenching. The essential difference between this formulation and the others of this group was that I7 contained ferric oxide powder. This material was added in order to darken the coating and to increase hiding power. The hiding strength of ultramarine is of low order. The original color of I7 was a grayish black. When heated, the coating on these panels turned a lavender color. This change in color, alone, indicates the unsuitability of ferric oxide as a pigment for this problem.

None of the other formulations evidenced any failure other than fading after being heated for seventy-two hours. A few panels were made up of I3 over a base coat of H21. These panels did not give evidence of fading. Formulations containing only five parts of ultramarine blue had much poorer covering ability than those containing ten parts of ultramarine blue.

None of the blue coatings so far discussed were close to the desired color. All were much too light. A number of formulations were prepared incorporating lampblack with ultramarine blue in a silicone-glyceryl phthalate vehicle. As shown in Table VIII, on heating these coatings to 800° F., the lampblack reacted with the oxygen in the air, and thus its effect was removed from the coating. In those cases where the formulation was not applied over a base coat, some ultramarine blue also faded out. Thus the base coat not only gives corrosion resistance, but also gives better adhesion to the finish coat.

TABLE VIII - FORMULATIONS CONTAINING LAMPBLACK

Sample Number	Pigment			Color Before Heating at 800° F.	Color After Heating at 800° F.
I10	10 U.B.	0.4 L.B.		Deep Blue Black	Ultramarine
I11	5 U.B.	0.4 L.B.		Deep Blue Black	Ultramarine
I12	10 U.B.	0.8 L.B.	1 Al	Gray	Light Blue
I13	10 U.B.	0.2 L.B.		Dark Blue	Ultramarine
I14	10 U.B.	0.1 L.B.		Dark Blue	Ultramarine
I15	5 U.B.	0.1 L.B.	2.5 Zn	Dark Blue	Ultramarine

Note: Vehicle in all cases was 10 DC802 15 G.P.

L.B. - Lampblack

All quantities are parts by weight.

Copper-bronze powder was tested in an attempt to obtain a dark topcoat. Along with this pigment, chromium oxide green was also evaluated. It had been observed that the color standard had a greenish cast. Chromium oxide green will withstand temperatures of refractory intensity (34), and has a green shade. Thus, along with the copper-bronze powder, the applicability of chromium oxide green to the problem could be determined. Before formulating the finishes, the copper-bronze powder was heated in a crucible over a Meker burner for two hours. This heating converted the powder to a dark oxide. The formulations of Table IX were then prepared.

TABLE IX - FORMULATIONS CONTAINING COPPER-BRONZE OXIDE

Sample Number	Pigment			Vehicle	
I16	10 U.B.	5 C.B.O.		10 DC802	15 G.P.
I17	5 C.O.B.	1 C.B.O.		10 DC802	15 G.P.
I18	5 C.O.G.	2 C.B.O.		10 DC802	15 G.P.
I19	2 C.O.G.	2 C.B.O.	3 Zn	10 DC802	15 G.P.
I20	2.5 C.O.G.	5 C.B.O.		10 DC802	15 G.P.
I21	1 C.O.G.	5 C.B.O.		10 DC802	15 G.P.
I22	0.5 C.O.G.	5 C.B.O.		10 DC802	15 G.P.
I23	1 C.O.G.	5 C.B.O.	1 Al	10 DC802	15 G.P.

Note: C.B.O. - Copper bronze oxide powder

C.O.G. - Chromium oxide green

All quantities are parts by weight

All of the formulations listed above were heated in the muffle furnace for eighteen hours. This treatment was followed by a quench in cold water. None of these panels failed. While coatings containing a large ratio of copper bronze oxide to chromium oxide green were darker than those with lower ratios, the desired darkening was not obtained.

The preparation of a uniform black copper-bronze oxide was difficult. Some commercially prepared copper oxide was obtained and a group of formulations compounded, I36, I37, I38, I39, I46, I47, I48. Extensive testing of panels coated with these formulations was not carried out. Particle size of the copper oxide powder was too large, and gave a rather rough coating possessing poor hiding characteristics. The copper oxide powder was screened, and only that powder which passed a standard 200 mesh screen was used in formulations. Larger particles were reduced to 200 mesh on the roll mixer using steel balls in a paint can. The formulations of the following table were made to test the suitability of this fine copper oxide powder to the problem.

TABLE X - FORMULATIONS CONTAINING 200 MESH BLACK COPPER OXIDE POWDER

Sample Number	Pigment			Vehicle	
I49	1.5 U.B.	10 CuO	5 Zn	10 DC802	15 G.P.
I50	1.5 U.B.	10 CuO	2 M.M.	10 DC802	15 G.P.
I51	1.5 U.B.	15 CuO		10 DC802	15 G.P.
I56	1.5 U.B.	10 CuO		30 Phenolic	

Note: CuO - Black copper oxide powder

All quantities are parts by weight

Using the above formulations a much smoother finish was obtained than when unscreened copper oxide was used. It was also found that prior to heating, the test panels approached the desired color. Furthermore, these coatings passed heating, quench, and bend tests. Facts which caused discontinuance of work with black copper oxide powder are: (a) the coatings changed to a gray color on heating, and (b) the coatings exhibited poor adhesion.

In searching for a suitable black pigment, manganese dioxide was investigated. The following formulations were tested in this phase of the investigation.

TABLE XI - SILICONE-GLYCERYL PHTHALATE FORMULATIONS CONTAINING MANGANESE DIOXIDE

Sample Number	Pigment				Vehicle	
I28	5 Zn	2 C.O.G.	0.1 MnO2		10 DC802	15 G.P.
I29	5 Zn	2 C.O.G.	0.5 MnO_2		10 DC802	15 G.P.
I30	5 Zn	2 C.O.G.	2 MnO_2		10 DC802	15 G.P.
I31	5 Zn	1 C.O.G.	2 MnO_2		10 DC802	15 G.P.
I32	5 Zn	1 C.D.G.	4 MnO_2		10 DC802	15 G.P.
I33	5 Zn	1 C.O.G.	4 MnO_2	1 U.B.	10 DC802	15 G.P.
I34	2.5 Zn	0.5 C.O.G.	4 MnO_2	1 U.B.	10 DC802	15 G.P.
I35		1.C.O.G.	4 MnO_2	1 U.B.	10 DC802	15 G.P.
I40	3 Zn		4 MnO_2	2 U.B.	10 DC802	15 G.P.
I41	3 Zn		4 MnO_2	1 U.B.	10 DC802	15 G.P.
I42	3 Zn	0.5 C.O.G.	4 MnO_2	2 U.B.	10 DC802	15 G.P.
I43	3 Zn	0.5 C.O.G.	4 MnO_2	1 U.B.	10 DC802	15 G.P.

Note: All quantities are parts by weight

By using manganese dioxide, a color was obtained which, prior to heating, approached that desired. This color was not stable. Upon being heated all coatings acquired a grayish cast. It was observed in addition, that adhesion could not be improved by varying the manganese dioxide - zinc ratio. The adhesion of all formulations was poor. They did, nevertheless, withstand a temperature of 800° F.

Since none of the black or dark gray pigments thus far studied had shown any promise, further investigations were made. Literature studies indicated that graphite might be suitable in these coatings, and formulations were prepared containing graphite. In these formulations, the graphite exhibited extremely poor wettability. As a result, it was decided to grind this pigment into DC802 silicone, on a roll mill, (Figure 3), prior to addition of other pigments. The graphite was ground into the silicone varnish in a one to one weight ratio. After several passes through the mill, the graphite still appeared poorly wet, and work with this pigment was discontinued.

Limited success had been obtained in preparing a coating of the desired characteristics using ultramarine blue, chromium oxide green, and various black and gray pigments. A completely new type of colored pigments was therefore investigated. These pigments were chromium, cobalt, and manganese aluminates. These pigments, in addition to their availability in the desired colors, were all of similar chemical structure. It was believed that they would be mutually compatible and would

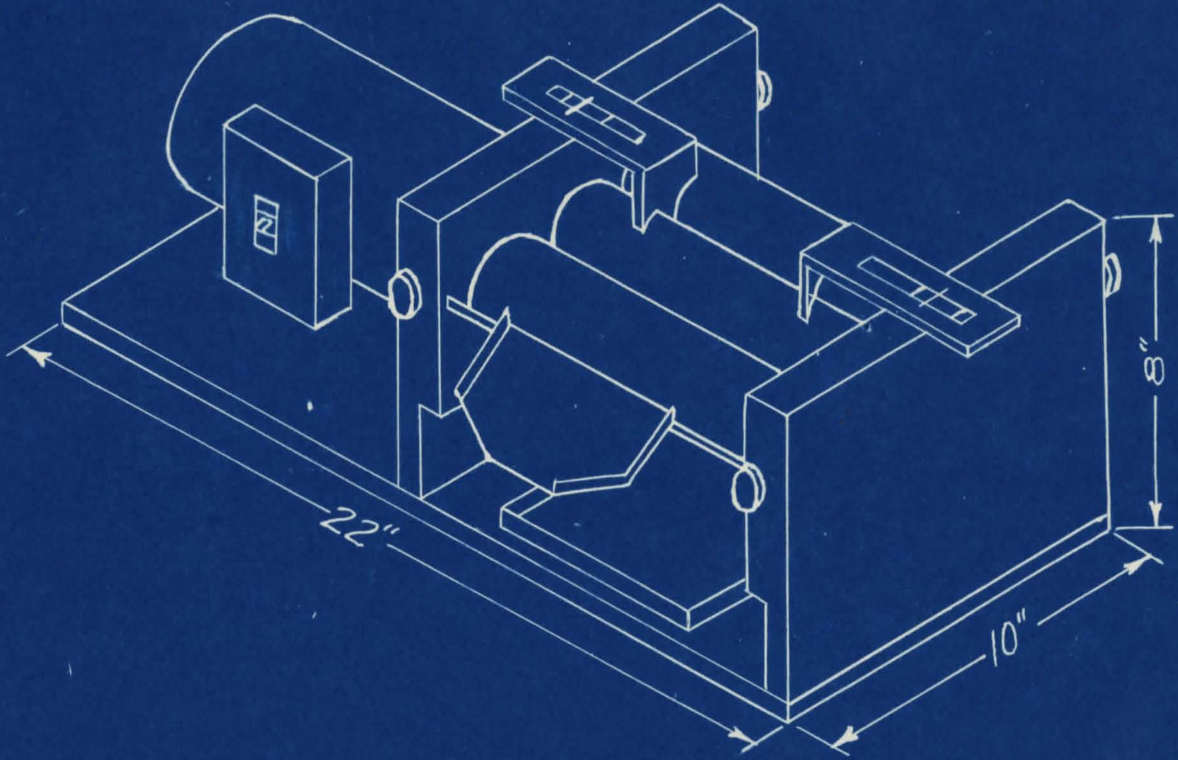

FIGURE 3 ROLL MILL

give the desired color match. Moreover, since they were structurally similar, color adjustments in the coating could be made at any time without materially altering physical properties of the coating.

The first step in using these pigments was determinding the approximate pigment ratio of the various colored oxides needed to obtain a suitable color match. The pigments were ground together using a glass plate and muller. DC802 silicone was used as the binding medium. A near perfect color match was obtained with the following pigment weights.

0.16 g.	Black Oxide 3900 -	0.35 g.	F-3203 Blue
0.12 g.	F-3630 Green	3.9 g.	DC802

This formulation was brushed on a panel over a base coat of H21. The panel was cured and heated in the muffle furnace at 800° F. for sixty-five hours. Upon the removal of the panel from the furnace, the color of the formulation was found to have remained the desired shade. By using the pigment ratio given above, and by bearing in mind the fact that the percent by weight of total pigment in a formulation should be approximately twenty-five percent, a series of possible top coat formulations was prepared.

TABLE XII - TOP COATS CONTAINING MINERAL OXIDE PIGMENTS

Sample Number	Pigment				
I69	2 Black	4.5 Blue	1.5 Green		
I70	1.15 Black	2.5 Blue	0.75 Green		
I72	4 Black	4.5 Blue	1.50 Green	6 Al	
I73	4 Black	4.5 Blue	1.50 Green	3 Al	
I74	4 Black	4.5 Blue	1.50 Green	5 T.F.	
I75	4 Black	4.5 Blue	1.50 Green	5 N.F.	
I76	4.5 Black	2.0 Blue	1.50 Green		
I87	2 Black	4.5 Blue	1.50 Green	0.5 W.M.	0.25 Al
I88	2 Black	4.5 Blue	1.50 Green	0.5 W.M.	0.25 Al
I89	2 Black	4.5 Blue	1.50 Green	0.5 W.M.	
I90	2 Black	4.5 Blue	1.50 Green	0.5 W.M.	0.25 Al

Note: With the exception of 189 and 190 all formulations in the preceding table were made in a 10 DC802 - 15 G.P. vehicle. Formulations 189 and 190 were made in a 25 DC802 vehicle

T.F. - Tin flake

N.F. - Nickel flake

W.M. - Waterground mica

All quantities are parts by weight

Formulation I69 was tested on panels as a single coat system, and also in conjunction with H21 as a two coat system. These panels withstood fifteen hours exposure at 800° F., quenching, and bending. Adhesion was not quite as good as desired. After 200 hours in the accelerated weathering unit panels of the two coat system H21-I69 showed only slight washing on the unbent portions. Thirty hours in the salt spray chamber had no additional effect.

Formulation I70 was an attempt to increase adhesion by addition of zinc. This coating did possess better adhesion. However, the color was too light, and the coating showed a washing tendency when weathered.

Formulations I72 and I73 were attempts to pigment the basic topcoat H1. To minimize the leafing characteristics of the aluminum, and thus improve hiding power, these formulations were permitted to stand fifteen hours before spraying. This standing had little if any effect on the leafing characteristics of the aluminum. A sample of degreased aluminum was obtained, and tried in these formulations. The hiding power of this aluminum was also very great. Attempts to reduce the hiding ability of aluminum were then discontinued.

In further attempts to increase adhesion of the blue coating, tin and nickel flake were tried in formulations I74 and I75. These materials neither increased adhesion nor permitted attainment of a good color match.

Several formulations, I87 through I90, were prepared using 325 mesh waterground mica and small amounts of aluminum. Waterground mica is said to offer leafing characteristics similar to those of aluminum (35). However, mica does not hinder the hiding ability of the other pigments. Mica was therefore added to increase the adhesion, and impart better weathering characteristics to the coating. Formulations containing small amounts of aluminum were too light in color. Those containing only silicone as vehicle did not cure properly at the desired temperature.

Formulation I69 was applied to the pipe stand. The coating did not fail on those portions of the pipe which were at 800° F. or less. It was observed, however, that there was a color gradient along the pipe. The hotter portions of the pipe were lighter in color. A coating with the same pigmentation as I69 but with a vehicle consisting entirely of DC802 silicone was applied to the pipe stand. This coating did not cure under the infra red lamp. There was no color gradient on this pipe after it was heated at 800° F., and the coating did not fail below 800° F. Both pipes had been previously sprayed with H21 base coat.

SINGLE COAT SYSTEM

Efforts were made to modify the original two coat system, H21-H1, to obtain a single coat system.

Tests were run using an alkyd resin (Beckosol 1316) and a melamine formaldehyde resin (Melmac 245-8), both with and without addition of DC802 silicone. The results are shown in Table XIII.

TABLE XIII - HEAT RESISTANT QUALITIES OF VARIOUS RESINS

Vehicle	15 Hrs. 475° F.	15 Hrs. 575° F.	15 Hrs. 675° F.	15 Hrs. 800° F.
DC802	Good	Good	Good	Failed
Melmac 245-8	Failed	--	--	--
Beckasol 1316	Good	Fair	Burned	--
15 Melmac-10 DC802	Good	Good	Good	Failed on Cooling
15 Beckasol-10 DC802	Good	Good	Burned	--

Note: All quantities are parts by weight

Stainless steel flake was also tested in attempting to prepare a satisfactory one-coat system. The formulations used are shown in Table XIV. All coatings were heated at 800° F. for eighteen hours, and quenched in cold water. There were no failures. The coatings all corroded after twenty-four hours in the salt spray cabinet. The corrosion was more marked in the case of those coatings containing a phenolic vehicle than in those containing silicone-glyceryl phthalate vehicle. This correlation was particularly noticeable after forty-eight. hours exposure in the salt spray cabinet.

TABLE XIV - FORMULATIONS CONTAINING STAINLESS STEEL

Sample Number	Pigment		Vehicle	
I65	6 S.S.		30 Phenolic	
I66	5 S.S.	1 Al	30 Phenolic	
I67	6 S.S.		10 DC802	15 G.P.
I68	5 S.S.	1 Al	10 DC802	15 G.P.

Note: S.S. - Stainless steel

All quantities are parts by weight

SUMMARY AND CONCLUSIONS

Two satisfactory two coat systems were obtained. These systems will withstand temperatures higher than 800° F., and do not peel off when quenched in cold water from that temperature. The coatings will bend satisfactorily over a one-half inch mandrel. The base coat for both systems is the same. It is a zinc-aluminum-cadmium pigmented carbonizing phenolic varnish. For one top coat an aluminum-zinc pigmented silicone-glyceryl phthalate vehicle is used. For the other, an aluminum pigmented silicone-glyceryl phthalate vehicle is used. The first top coat does not gel in the can as rapidly as the second. The heat resistant characteristics of the second top coat are superior.

The coatings do not require air-drying, but must be baked under an infra red lamp to obtain proper curing. The better system (that with the aluminum pigmented top coat) has good weather resistance and will withstand the action of oil and toluene at 300° F. and 200° F. respectively.

Coatings which have micronized mica and/or asbestine in either of the two coats exhibit poorer adhesion than those without these materials.

Iron blue, Chinese blue, bronze blue, phthalocyanine blue, and indanthrene blue are all unsatisfactory for a high heat resistant blue coating. The only standard blue pigment which was found to be capable of withstanding an 800° F. temperature, was ultramarine blue. Chromium oxide green withstood the 800° F. temperature. No standard black pigment was found which would serve satisfactorily at this temperature.

Various mineral oxide pigments were obtained which permitted blending to the desired shade of blue. A reasonably satisfactory coating is obtained when these pigments are used in a silicone-glyceryl phthalate vehicle. The coating should be applied over the previously mentioned base coat. Further work is necessary to increase adhesion of this coating to the base coat, as this particular property is not yet as desired.

Attempts were made to modify the two coat system so that a single coating would satisfy the requirements. Various resins other than silicones and glyceryl phthalate were evaluated. These resins included melamines and alkyds. No satisfactory single-coat system was obtained.

A number of proprietary heat resistant coatings were evaluated. All these coatings failed on heating and/or weathering.

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APPENDIX

APPENDIX I

NOMENCLATURE

1. Asb. - Asbestine
2. Al - Aluminum paste
3. C.B.O. - Copper-bronze oxide
4. Cd - Cadmium powder
5. C.O.G. - Chromium oxide Green
6. CuO - Black copper oxide powder
7. DC801 - Dow Corning 801 silicone resin
8. DC802 - Dow Corning 802 silicone resin
9. G.P. - Glyceryl phthalate spar varnish
10. I.B. - Iron blue
11. L.B. - Lampblack
12. M.M. - Micronized mica
13. N.F. - Nickel flake
14. Phenolic - Heat reactive phenolic varnish
15. S.S. - Stainless steel flake
16. T.F. - Tin flake
17. U.B. - Ultramarine blue
18. W.M. - Waterground mica
19. Zn - Zinc powder

APPENDIX II - FORMULATIONS CONTAINING BLACK COPPER OXIDE POWDER

Sample Number	Pigment			Vehicle	
I36	5 CuO	0.50 C.O.G.	2.0 U.B.	10 DC802	15 G.P.
I37	5 CuO	0.25 C.O.G.	1.0 U.B.	10 DC802	15 G.P.
I38	5 CuO	0.50 C.O.G.	1.0 U.B.	10 DC802	15 G.P.
I39	5 CuO	0.25 C.O.G.	0.5 U.B.	10 DC802	15 G.P.
I46	10 CuO		1.5 U.B.	10 DC802	15 G.P.
I47	20 CuO		3.0 U.B.	10 DC802	15 G.P.
I48	10 CuO	0.75 C.O.G.	1.5 U.B.	10 DC802	15 G.P.

APPENDIX III - FORMULATIONS CONTAINING GRAPHITE

Sample Number	Pigment		Vehicle	
I57	0.4 Graphite	5.0 U.B.	30 Phenolic	
I58	0.4 Graphite	5.0 U.B.	10 DC802	15 G.P.
I59	4.0 Graphite	1.5 U.B.	30 Phenolic	
I60	4.0 Graphite	1.5 U.B.	10 DC802	15 G.P.
I61	1.0 Graphite	5.0 Zn	30 Phenolic	

Note: All quantities are parts by weight

APPENDIX IV

PROPRIETARY COATINGS TESTED

1. Thurmalox #10 - Withstands temperature of 800° F. Poor adhesion after heating. After heating, panels rusted within twenty-four hours in a water mist.
2. Sherwinn-Williams Heat Resistant Aluminum - Withstands temperature. Rusted after twenty-four hours in a water spray.
3. DuPont Heat Resistant Aluminum - Will not withstand 800° F. Poor weathering.
4. Yarnall Industrial Finish - Withstands 800° F. Poor weathering.
5. Glidden White Heat Resistant Paint - Failed at 800° F.
6. Permite 1100 - Failed at 800° F.
7. Permite 1001 - Blistered at 800° F. - Rusted in twenty-four hours in weatherometer.
8. Socony-Vacuum Heat Resistant Paint - Withstands 800° F. - Poor adhesion.
9. "Kaycote" Heat Resisting Paint - Withstands 800° F. - Poor weathering.
10. "Mico" Heat Resisting Paint - Withstands 800° F. - Poor weathering.

APPENDIX V

COMMERCIAL MATERIALS USED AND THEIR MANUFACTURERS

1. Paste, Aluminum, Grade A, Army-Navy Aero. Spec.
   AN-TT-A-461a
   Contractor: U. S. Bronze Powder Wks. Inc., New York, N.Y.
2. Paste, Aluminum, 30 LN
   Reynolds Metals Co. Inc., Louisville, Ky.
3. Standard Zinc Dust - 22 (Horse Head Products)
   New Jersey Sales Co., New York, N. Y.
4. MD 201 Cadmium Powder
   Metals Disintegrating Co. Inc., Elizabeth, N. J.
5. MD 750 Nickel Flake
   Metals Disintegrating Co. Inc., Elizabeth, N. J.
6. MD 8601 Tin Flake
   Metals Disintegrating Co., Inc., Elizabeth, N. J.
7. Stainless Steel Flake
   Charles Hardy Inc., New York, N. Y.
8. Graphite - Fed. Spec. SS-G-659 Grade fine
   Joseph Dixon Crucible Co., Jersey City, N. J.
9. Micro Mica - 3000 Mesh
   English Mica Co., Spruce Pine, N. C.
10. Waterground Mica - 325 Mesh
    English Mica Co., Spruce Pine, N. C.
11. Black Copper Oxide Powder S.7256
    Merck and Co. Inc., Rahway, N. J.

12. Ultramarine Blue #3155

    C. J. Osborn Co., New York, N. Y.

13. Chromium Oxide Green #3102

    Kentucky Color Co. Inc., Louisville, Ky.

14. F-3203 Blue Oxide

    Ferro Enamel Corp. Inc., Cleveland, Ohio

15. F-3630 Green Oxide

    Ferro Enamel Corp. Inc., Cleveland, Ohio

16. Black Oxide 3900

    B. F. Drakenfeld & Co. Inc., New York, N. Y.

17. Varnish, Spar, Glyceryl Phthalate, Spec. AN-TT-V-116

    Beckwith Chandler Corp., Newark, N. J.

18. Varnish, Heat Reactive Phenolic Clear - VG5293

    E. I. duPont De Nemours & Co. (Inc.), Wilmington, Delaware

19. DC802 Silicone Resin

    Dow Corning Corp., Midland, Mich.

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VITA

Arthur Hoff Isaacs son of Louis Isaacs, and Sarah (Lewin) Isaacs was born the first day of September 1924. He received his elementary education at Longfellow School of Louisville, Kentucky, and his junior high school education at Highland Junior High School of the same city. His secondary education was obtained at The Louisville Male High School, from which he was graduated in June, 1942. He began the study of chemical engineering at the University of Louisville and pursued his studies there until his enlistment in the United States Navy in June, 1944. After a period of two years in the Navy he returned to the University of Louisville to continue his studies in chemical engineering, and received the degree of Bachelor of Chemical Engineering in September, 1947. He further pursued his studies at the Graduate School of the University of Louisville from which institution he received the degree of Master of Chemical Engineering in June, 1949.