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SURFACE FINISHING OF STAINLESS STEEL
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tion of Pennsylvania
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This application is a continuation-in-part of application Serial No. 48,573, filed August 10, 1960, and of application Serial No. 173,141, filed February 14, 1962, both now abandoned.

This invention relates to the improvement of the surface of stainless steel, and is particularly directed to stainless steel surfaces which are resistant to marring, marking, fingerprinting and scratching, and resistant to heat tinting at elevated temperatures.

In modern architectural design of buildings, and particularly office and business buildings in metropolitan areas, there is an increasing demand for stainless steel building panels for exterior applications. This metal possesses the desired and required corrosion resistance for longtime outdoor atmospheric exposure, plus the required formability and the desired mechanical strength. Also, this metal exhibits a particularly desired bright and lustrous attractive metallic appearance that may be modified by surface treatment to effect a multitude of reflective or of non-reflective surfaces that are ideal for either indoor or outdoor building panel applications. In consequence, it may be seen that stainless steel is an ideal material from which one may produce building panels. Such steel does, however, possess one major drawback that in many instances causes those in charge of selecting materials for architectural design to hesitate in making this selection. Such drawback is the tendency of a stainless steel to show scratches and marks that invariably are imposed on any metal surface being subject to installation for applications such as architectural building panels. It is well known that marks, such as scratches and gouges, effected during installation are negligible and are not readily observed after installation of most metal panels formed of metal other than stainless steel, but similar scratches or gouges on stainless steel panels are readily observable after installation and detract from the appearance of the stainless steel. Such scratches and marks may not be removed after installation of stainless steel panels by any known commercial means without destroying the continuity of a uniform appearance of such panels.

In addition, stainless steel surfaces are highly susceptible to fingermarks placed on the steel surface by workmen during installation. Such marks are caused by the natural skin oils that exist on human hands. Additionally, black dirt or greasy deposits are commonly, though inadvertently, smeared onto the surface of stainless steel during installation. Such fingermarks and smears are not easily removed from a stainless steel surface. No modern-day cleaner has been perfected that will completely remove such fingermarks or smears so as to leave an unmarred, attractive stainless steel surface.

Such surface phenomenon as set forth above has severely restricted the use of stainless steel, particularly for building panel applications where a roughened or buff finish is desired to minimize reflectivity. The scratch and mark susceptibility of stainless steel not only detracts from its use as building panels, but also is detrimental in other applications where the appearance of the steel surface is of importance. For example, the fingermarks and smear appearance detract from the application of stainless steels in the dairy industry, since cleanliness is of primary importance in this industry, and on some stainless steel surfaces such marks are virtually impossible to remove with

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cleaners, and only gradually disappear after many repeated cleansing operations over long periods of time.

It has now been found that an extremely thin, substantially invisible silicate coating on stainless steel will so alter the mechanical scratch and mark susceptibility of the coated surface of such steel as to render the appearance of scratches and marks negligible in comparison to similarly scratched and marked uncoated stainless steel. Also, it has been found that the thin silicate coated stainless steel is resistant to fingerprints and smear marks and that when such coated steel is smeared with a greasy substance, the smear may be easily removed without leaving a marred or blemished surface.

Accordingly, it is a principal object of this invention to provide a stainless steel surface that exhibits improved resistance to the adverse appearance of mechanical scratches and marks.

It is also an object of this invention to provide a stainless steel surface that is resistant to fingermarks or smear marks.

A still further object of this invention is to provide an improved surface to stainless steel from which fingermarks and smears may be totally removed by wiping with a cloth.

Yet an additional, more general object of this invention is the provision of a stainless steel surface which has improved resistance to marring, marking, fingerprinting and staining.

Further, the application of stainless steel to the market of home appliance and other uses where the stainless steel is subjected to elevated temperatures up to about 1000° F. has been greatly hindered by the propensity of stainless steel to oxidize, causing heat tinting of the exposed surfaces at temperatures above 300° F. The resulting color from this heat tint is detrimental to the appearance of the stainless steel, and in some cases to its corrosion resistance. As a result, domestic appliances and parts thereof, such as stove burner rings and "spiders" oven interiors, toasters, clothes dryer interiors, waffle irons, sandwich grills, coffee pots, fry pans, etc., are now conventionally made of chrome-plated steel or aluminum to prevent heat tint where used at the temperatures commonly employed by such appliances. Usually heat tint forms within the range of from about 300° F. to 1000° F. and the stainless steel employed in the utilization of the aforementioned appliances is usually subjected to temperatures within this range. Other applications of stainless steel where a heat tint type of oxidation is a problem include reflectors for high power lights. Chromium flashing has been successfully used on stainless steel to retard heat tinting at temperatures in the range of 700° F. and below, but this is expensive and is limited in the degree of protection afforded, especially at higher temperatures. From the above it is apparent that a practical treatment which would give protection against heat tinting and oxidation resistance constitutes a significant advance in the stainless steel art as it relates to the appliance field.

It has been found that an extremely thin coating of an alkali-metal silicate on the visible surface of stainless steel in such articles will prevent heat tinting at temperatures between 300° F. and 1000° F. and even higher.

Accordingly, it is a further principal object of this invention to provide a stainless steel surface that exhibits improved resistance to the adverse appearance of heat tint when subjected to temperatures in the range of from about 300° F. to 1000° F.

It is also an object of the present invention to provide a stainless steel for use in the fabrication of appliances such as stoves, ovens, toasters, etc., with a coating that will not detract from the luster normally provided by stainless steel, but which will prevent the formation of

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heat tint on the stainless steel surfaces when such articles are subjected to intermittent or prolonged heating at temperatures in the range of from about 300° F. to 1000° F. as is ordinarily the case.

A further object of the present invention is to provide an improved surface to stainless steel which will not detract from the beauty of the appearance of stainless steel, but which will resist heat tint within the temperature range of from about 300° F. to 1000° F.

These and other objects, together with a fuller understanding of the invention, may be had from reference to the following specification and claims.

In general, the present invention embodies a stainless steel member having a surface that has been coated with a thin (.001" max.), substantially water insoluble, alkali metal silicate coating. In the case of building panels and other architectural members, the surface finish generally will be no brighter than a No. 4 sheet finish, and will exhibit improved resistance to marring, marking, fingerprinting and staining, and in the case of stainless steel members used where heat tinting is likely to occur, the members will generally have a surface finish that is brighter than a No. 4 sheet finish and the surface will resist the tendency toward heat tinting at temperatures between 300° F. and 1000° F. In any event, the present invention is of significance with any exposed stainless steel surface where surface appearance is of importance and these surfaces can therefore be considered decorative surfaces.

With respect to the architectural members, the improved coated stainless steel surface of the present invention is almost identical in appearance with non-coated panels having the same surface finish. In other words, the extremely thin coating is transparent and substantially invisible, inasmuch as it cannot be ascertained as even being present under ordinary inspection. However, when the coated steel is marked or scratched with a sharp metallic edge, the difference in appearance from that of an uncoated panel similarly scratched is readily observable. The uncoated steel is marred or gouged or scratched, and such a defect is readily apparent, even to a casual observer. On the other hand, a similar scratch or gouge on a coated article is not so readily observed, although a close inspection will show some damage to the surface when viewed under the proper light, at the proper angle and at relatively short distance; however, such a defect will not be apparent to a casual observer, and without closely inspecting the panel he will not be aware of its presence.

The silicate coating of the present invention is particularly significant and useful in the architectural field when applied to finishes that are ordinarily employed for building panels. These finishes are generally of the buff or dull type when compared to the bright and lustrous finishes that can be obtained on stainless steel. Buff or dull, non-reflecting surfaces are ordinarily necessary in the field of outdoor building panels to prevent undue reflection of the sun, and such finishes are sometimes applied to the stainless steel panel by passing the panel through rolls that have been sand blasted or shot peened or otherwise treated so as to impose, by embossing, the buff imprint on the stainless surface. For the aforementioned reasons, it is unlikely that outdoor building panels exhibiting a brighter finish than a No. 4 sheet finish would be used for architectural panels. By employing the coated panels of the present invention, installation can be accomplished in the usual fashion without fear of gouged or scratched surfaces in that such defects are not readily observable on the panel finish. In addition, few, if any, fingermarks or grease smudges will be observable on the stainless steel surface, and if any should be present and detract from the panel's appearance, they may be completely removed by simply wiping with a wet cloth or paper towel. Although buff or dull surface finishes having a reflectivity not greater than a No. 4 sheet finish may

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in some instances be used in applications where the stainless steel may heat tint, by and large the majority of such applications require a bright finished stainless steel surface greater in light-reflectivity than a No. 4 sheet finish, and hence the application of the silicate coating for protection against heat tinting is primarily directed toward bright finished stainless steel. In this instance, also, the surface of the stainless steel is coated with a thin (not greater than .001") substantially water insoluble alkali metal silicate coating. So-coated, the surfaces of such articles reveal vastly improved resistance to the heat tinting when subjected to temperatures of from about 300° to about 1000° F. The improved coated stainless steel surface is substantially identical to a similar surface of a non-coated article having the same surface finish. In other words, the extremely thin coating presently applied is transparent and invisible in that it cannot be ascertained as even being present under ordinary inspection. However, when coated stainless steel articles and uncoated stainless steel articles are subjected to temperatures in the heat tinting range, the difference is readily observable; the uncoated articles acquire the characteristic heat tint color, whereas the coated articles remain unchanged in surface appearance. The silicate coating utilized in the present invention is particularly significant and useful for resisting heat tinting when applied to finishes that are ordinarily employed in the manufacture of various component parts of appliances such as stove burner rings, spiders and medallions (round members disposed in the center of the coils of electric burners). Spiders are three-pronged members with circular centers used to support burners, and burner rings are the bright rings surrounding the burners. The coated stainless steel of this invention may also be employed as the inside panels of an oven, on a toaster, and other similar applications. Such finishes are generally bright and lustrous as compared to the generally dull and buffed finishes employed in the architectural field.

For the purpose of this invention, any silicate compound that is soluble in water may be employed for the silicate coating. However, for all practical purposes soluble silicates are limited to the alkali metal silicates, which are the only silicates soluble to any great extent in water. The term "alkali metal silicate" includes such materials as lithium, potassium and sodium; however, as is well known, sodium and potassium are the only such materials that are readily available in commercial quantities, and of the two, sodium silicate is far less expensive and far more readily available. However, since the invention is operable with any of these silicates, they are all included within the scope of the present invention, but for the purpose of illustrating the invention, specific reference will be made herein to the use of sodium silicate, commonly known as water glass.

The sodium silicates utilized in this invention are water soluble solids usually formed by fusion of sand and alkali metal carbonates. The resulting amorphous material may vary widely in its metal-silicon-oxygen ratio; for this reason, the compositions of silicates are usually expressed as $\text{SiO}_2:\text{M}_2\text{O}$ ratios (where M_2O is the alkali metal oxide). Some examples of sodium silicate are as follows:

Name	Formula	$\text{SiO}_2:\text{Na}_2\text{O}$ ratio
Sodium orthosilicate.....	Na_4SiO_4	1:2
Sodium metasilicate.....	Na_2SiO_3	1:1
Sodium disilicate.....	$\text{Na}_2\text{Si}_2\text{O}_5$	2:1
Commercial water glass.....		3.3:1
Others approaching ratio of.....		4:1

The ratio of about 4:1 is critical. Above this, the silicate is no longer adequately water soluble, and, in fact, the difficulty of dissolving the glasses in water increases with the ratio below the critical point so that steam may be used to dissolve those above the disilicate. In solution, these compounds exist in ionic form with the presence

of polysilicate ions in the case of the higher ratio silicate such as water glass. Such ions with molecular weights of 200-300 are probably unordered arrangements consisting of both 4 and 6 coordinated silicon atoms in a roughly spherical structure. In a sense, they are semi-colloidal in nature with high viscosity, but distinguish from colloidal silicates which are inoperative for the purpose of this invention.

When thin layers of such solutions are allowed to evaporate, a substantially transparent and invisible film or coating is formed. The thickness of such a coating is critical in that if the coating in its dried state is permitted to exceed about .001" in thickness, the coating becomes visible and interferes with the natural appearance of the stainless steel. Inasmuch as the attractive silvery appearance of the surface itself is one of its primary assets and may be the principal reason for the selection of stainless steel for the desired end use, its neat, clean appearance is highly desirable. Further, the neat, clean appearance of stainless steel is highly desirable in the food and dairy industry where stainless steel is extensively used for food handling equipment. Also, a coating even slightly thicker than .001" cannot be properly cured in that such thicker coatings will froth or bubble when cured at temperatures even as low as 300° F., as will be described presently.

After drying, which may take place at room temperature or at temperatures up to 212° F., the coatings are baked in order to cure or set them so as to render them substantially water insoluble. Coatings which have been dried at room temperature of up to 212° F., in fact, have good mechanical properties but are extremely water soluble. A substantial degree of insolubility of the silicate coating is attained by baking at about 300° F., although long-time immersion in water will gradually dissolve such coatings. As the baking temperature is increased, the solubility decreases until the coatings are substantially completely insoluble after baking at 480° F. Temperatures between 480° and 600° F. are preferred for curing, although temperatures up to 1250° F. may be used. It has been found, however, that temperatures greater than 600° F. may have some detrimental effects on the mechanical properties of certain grades of stainless steel. It has been found, however, that with coatings approaching the .001" max. thickness the baking temperature cannot greatly exceed 300° F. or there will be some tendency toward bubbling or frothing, and as the baking temperatures increase, thinner coatings are required in order to prevent this phenomenon of frothing or bubbling. At the thickness of approximately .00005", which is a thickness readily attainable commercially on both dull and bright panels, baking temperatures as high as 1250° F. have no adverse effects on the coating and no frothing or bubbling occurs. Baking times of thirty minutes or less are sufficient, and the time at temperature may be as little as five to ten minutes. The exact effect of baking is not known. Possibly the temperatures involved may cause polymerization of the already partially polymerized (molecular weight 200-300) polysilicate ions to form more —Si—O—Si— bonds. However, it is possible that the reaction simply involves removal of physically and chemically absorbed water.

The solution employed to effect the coating may be sprayed upon the surface of the stainless steel or the stainless steel may be dipped into such a solution so as to effect the desired films. Additionally, the silicate coatings may be applied to strip or sheet by being rolled on. Such process is a commercially known coating means wherein the coating material is constantly applied to the surface of a roll which is, in turn, rolled over the surface of the metal strip or sheet.

The concentration of alkali metal silicate present in the aqueous solution is not critical in that any concentration may be employed that will effect such a film, and merely any amount from a trace to saturation will effect a film

which varies in thickness in accordance with the strength of the solution employed. For practical purposes, however, it is desirable to employ a solution containing at least about .1%, by weight, of the alkali metal silicate to effect a useful coating. It is entirely feasible, however, to employ aqueous solutions that are saturated with the silicate compound. The temperature of application is also unimportant so long as at least about .1% of the silicate is present in the water solution.

In applying the silicate coating it is preferable, though not essential, to add to the vehicle or coating solution a small quantity of a wetting agent. Such wetting agent may be any of the commercially available water soluble wetting agents that are compatible with the silicate solutions. The effect of such additions is to aid in securing a smooth and even coating on the surface being silicate coated. Excellent results have been obtained by employing 10 ml. of a 10%, by weight "Aerosol AY" solution with a 75 ml. sodium silicate solution (technical, 40-42° Bé.). "Aerosol AY" is the trade name of the American Cyanamid Company, 30 Rockefeller Plaza, New York 20, New York, and is designated to identify solutions containing as the active ingredient diamyl sodium sulfosuccinate. Another commercially available wetting agent that has been satisfactorily employed is a modified sodium lauryl sulfate sold under the trade name of "Wetanol" by the Glyco Products Company, Empire State Building, New York, New York.

The term stainless steel, as applied in the present specification, is intended to include all the steels classified by the American Iron and Steel Institute as being standard grades of stainless steel. These include the Type 400 series stainless steels that contain chromium in amounts of from about 10%, by weight, to about 30%, by weight, and generally less than 1% carbon, such as AISI Types 410 and 430 and additionally the AISI Type 300 series which contain, in addition to Cr and C, a nickel content of from 6 to 30% which renders the steel structure "austenitic," such as AISI Type 301, 302 and 304, and the 200 series steels which contain not only nickel in amounts of 1 to 10%, but also up to about 30% Mn and .60% N₂ as additional austenitizers. Such various stainless steel analyses may contain additionally, as impurities or alloying ingredients, small amounts of P, S, Cu, Mo, Se, B, Be, Co, W, Ti, Cb, Ta, V, Zn, Al, Si, rare earths, etc. All stainless steels, however, contain chromium within the range of from about 10% to 30% and carbon up to about 1%. The Cr content in every instance is the element that primarily effects the essential property of oxidation and corrosion resistance, and consequently the article of the present invention may be broadly said to be composed partly of a steel that consists essentially of carbon in an amount up to about 1%, chromium from 10 to 30% and the balance iron.

FORMULATION OF COATING SOLUTION

Formulation of the coating solution involves an alkali metal silicate, a wetting agent and water. I have found the sodium silicate solution to be particularly satisfactory and have specifically employed Fisher Scientific Company or Philadelphia Quartz Company's "N" Grade. Fisher's solution has 28.18% SiO₂, 8.22% Na₂O for a Na₂O:SiO₂ ratio of 1:3.43; Philadelphia Quartz's grade has 28.59% SiO₂, 8.50% Na₂O for a ratio of 1:3.36. No differences in coating quality or characteristics have been noticed. Normally this grade of sodium silicate solution has the following characteristics:

Percent Na ₂ O	8.90
Percent SiO ₂	28.70
Ratio, percent Na ₂ O:percent SiO ₂	1:3.22
Percent solids	37.6
Degrees Baumé (68° F.)	41.0
Specific gravity (68° F.)	1.394
Lbs. per gallon	11.6
Viscosity (centipoises)	180

The concentrated solution is referred to as 100% so that various dilutions used in coating herebelow refer to a volume-to-volume relationship e.g. 25% solution is 25% concentrated solution by volume and 75% water. To obtain percent solids, a value which is widely used in coating work, the percent solution is multiplied by .376. Solutions range from 10% to 100%, depending on method of application, type of surface and end use. The most common dilutions are 30% and 50%. Tap water may be used if necessary for dilution, but distilled water is preferred. The characteristics of various dilutions obtained from literature and/or experiments are given in Table I. These values should be taken as approximate because of batch-to-batch variations in the concentrated solution.

Table I

PROPERTIES OF COATING SOLUTIONS

Vol. percent sodium silicate solution in H ₂ O	pH	°Bé. ¹	Specific gravity	Viscosity, ² Zahn seconds	
				#2 Cup	#1 Cup
100	11.05	41	1.39	40	---
95	---	---	---	22	43
90	---	39	1.37	20	---
80	---	36	1.33	16	33
65	---	---	---	---	20
50	11.05	25	1.21	15	30
25	11.05	11	1.08	15	28
10	11.00	6	1.04	---	---
30	7.00	---	1.00	---	28

¹ By hydrometer at room temperature.

² Room temperature—no close control.

³ Dist. H₂O.

The addition of a wetting agent is not always necessary but it is desirable in general to insure the best possible wetting action at the metal surface. Alkaline silicate solutions are incompatible with many organic materials and this fact eliminates a considerable number of surface active agents commonly used in coatings. Several such agents have, however, been found to be compatible with the coating solutions. Several agents and their compatibility are listed in Table II.

Table II

SURFACE ACTIVE AGENTS (WETTING AGENTS)

Name	Active Ingredient	Type	Compatibility with Sodium Silicate Solutions
Aerosol A.Y.	Diamyl sodium sulfosuccinate	Anionic	+
Aerosol O.S.	Propylated naphthalenesulfonic acid	do.	—
Aerosol O.T.	Di (2-ethylhexyl) ester of sodium sulfosuccinic acid	do.	—
Laboratory Aerosol	Aerosol O.T. with mutual solvent	do.	—
Alkanol HCS	A sodium alkyl naphthalene sulfonate	do.	—
Tergitol Anionic OS	Sodium 2-ethylhexyl sulfate	do.	—
Triton 102	p-(Diisobutyl) C ₈ H ₁₇ (OCH ₂ CH ₂) ₂ OSO ₃ Na	Nonionic	—
Wetanol	Modified sodium lauryl sulfate	Probably anionic	+
Ethofat 0-15	Polyoxyethylene (15) glycol talloil ester	Nonionic	—
Marasperse C	Sodium lignin sulfonate	Anionic	+
Marasperse N	do.	?	+
Ethofat 242/25	Polyoxyethylene (15) glycol talloil ester	Nonionic	+

The coating solution should always be filtered before use, especially after addition of the wetting agent. The commercial solution often contains fine gelatinous particles, and the wetting agent will sometimes cause gelatinous precipitates to form in small quantities. Slight residual cloudiness in the solution will cause no harm, but it can be filtered to a completely clear liquid.

As indicated in Table I, viscosity very quickly reaches a low level with continued dilution, the "thinness" of water being very nearly reached at 80%, a concentration above that normally used for coating work. Therefore, in planning coating procedures, viscosity must be considered a non-variable. This leads to certain difficulties in application as discussed in the next section. Before leaving the subject of formulation, it is important to realize that sodium silicate solution should be kept above

the freezing point since some irreversible precipitation will occur if the solution is frozen. For the concentrated solution, 25° F. is the minimum temperature; dilute solutions should be kept above 32° F.

APPLICATION

Most of the standard paint application methods have been investigated for use with this coating and, given suitable conditions, nearly all have proved satisfactory. The following subsections will treat each method separately:

(1) Dipping

This method is useful on a laboratory scale because of its ease and rapidity, and the very uniform and repeatable coatings which it can yield. The procedure consists of immersing the metal in a bath of coating solution and withdrawing at a slow, uniform rate by means of a motor-driven cord or cable attached to the sample. The dry coating thickness is determined primarily by the concentration of solution, wet thickness being constant for constant viscosity and withdrawal rate, both of which can be considered nearly invariable in this system. Wet thickness in dip coatings is determined by a balance of forces between surface tension and viscosity on one hand, and gravity on the other. Withdrawal rate is such that essentially no excess solution is deposited on the surface and therefore no run-down occurs after the film is above the solution level. Using a 50% solution, a withdrawal rate of 3 inches per minute has been found to give excellent coatings on any vertical surface. Necessary precautions include prevention of vibration and the use of clean coating solution, especially at the surface. Floating foam, dirt, bubbles or dried particles of coating will be deposited on the metal surface and show up as imperfections.

Complex shapes, where the surfaces are at various angles to the vertical, can be coated if no surface is horizontal or very close to it. However, because different angles to the vertical will have different actual withdrawal rates even though the piece as a whole is withdrawn at one rate, the rate must be slow enough so that the most nearly horizontal surface will not run. For example, while vertical surfaces can be withdrawn at rates up to 6 inches per

minute, a bright surface at 45° from the vertical must be withdrawn at not more than 1 inch per minute. Rough surfaces can be withdrawn somewhat faster than those which are smooth and bright.

(2) Flow coating

The application of coatings by means of flowing the solution over the surface and allowing it to drain has become commercially important. There are many variations on this process, ranging from simply pouring the solution over the piece to complex equipment which delivers a metered flow either from low pressure orifices or in the form of a liquid "curtain." The only work done here has been by slowly pouring the coating solution (about 30% concentration) over the piece and allowing it to drain and dry. After baking, the process is re-

peated to give sufficient coating thickness. The coating so obtained is not really uniform, but this is usually not noticeable and results are satisfactory, especially on formed parts. A variation is the immersion of the part in solution, quick withdrawal and draining. This may appear to be a form of dip-coating, but the principle is actually closer to that of flow-coating. It has been possible to coat some intricately formed parts only by this method; it is therefore an important adjunct to the more generally applicable procedures such as spraying.

(3) *Spraying*

This is the most versatile method of application and one which can be widely used. The equipment being used in the laboratory consists of a P-JGA-5025 external mix spray gun, 704F air cap-fluid tip combination, and a P-KB-519 pressure feed attached cup, all of De Vilbiss make. (De Vilbiss Co., Toledo, Ohio.) A filter-pressure regulator combination controls air pressure which is usually used at 40 p.s.i. Material pressure is 1 to 5 p.s.i. with feed rate being controlled to give a fine mist which will thoroughly wet the surface, but will not cause running. The latter problem is especially prevalent with the very thin coating mixture, and it is sometimes preferable to build up the final dry coating thickness with two thin coats, the second applied after the first is dry. The coating dries very rapidly, especially when a gentle flow of air is directed at the surface. Care must be exercised to prevent excess thickness, which results, after baking, in a whitish appearance or bubbling if the thickness is in excess of .001". A wet thickness of .0005 to .001" (.5 to 1 mil) can be readily applied for a one coat system; this will yield about .05 to .1 mil dry thickness when using a 30% solution. Spraying has been done by hand, but automatic, multiple head spray equipment would do a more uniform and repeatable job, as well as being cheaper and faster.

The use of airless spray, electrostatic spray, or very fine, "misting" type, air spray heads should yield good spray results on a bright surface. Good results have also been obtained on such surfaces with the previously mentioned equipment, but it is difficult to get consistently acceptable coatings. Other than the spray equipment itself, clean surroundings (especially the air) and adequate ventilation are necessary for spray application. Clean-up is simplified with this coating, since the thinner is water, but equipment should be kept free of dry coating buildup because thick films are difficult to redissolve.

(4) *Spin coating*

This method is useful on a laboratory scale and embodies a variable speed motor which rotates a flat turntable. The sample to be coated is placed on the turntable and the motor started, which rotates the sample in the plane of its surface to be coated. Coating solution is poured from a beaker onto the surface of the spinning sample and the solution is evenly spread over such surface; one or more coatings can be applied in this manner to obtain the desired thickness. It has been found by utilizing this method of coating that dry coatings of predetermined preferred thickness of up to .0005" or even greater, up to the critical limit of .001", can be obtained.

(5) *Miscellaneous methods*

Other methods of coating such as brush or swab come to mind when considering application methods. Brush is not suitable for this coating, unless the solution is applied so thickly (and allowed to drain) that the flow coating principle is really being utilized. Good coatings on small, flat pieces have been obtained with swabs made up of a resilient, absorbent cloth such as felt stretched over a flat piece of wood and wet (but not soaked) method, but might be utilized in certain, specific cases where other methods are not possible.

(6) *Touch-up*

Because any agent which removes the clear coating locally would likely also change the appearance of the underlying stainless, it is quite doubtful if localized repair would ever be useful in service. However, since it is possible that occasional areas which for some reason were not coated might need repairing, experiments with spray and swab touch-up were performed. It was found that both methods would afford a passable, although not perfect, repair if the area was clean. Since stainless surfaces will heat tint slightly at baking temperatures if not protected by coating, the repaired area may show a slightly different color when the oxide is not first removed. For most repairs, spray works very well if the gun can be adjusted to give a small enough spray pattern. Small areas can be swabbed with a felt dauber of appropriate size. Thought must also be given to baking the repair, either by baking the whole piece or by using localized heating.

BAKING

When the sodium silicate film applied as described above is heated to temperatures in excess of 300° F., water is driven off and the coating becomes more and more water insoluble as time and/or temperature increases. From about 300° F. to 400° F., mostly physically absorbed water is given off; above 350° F. more strongly bound water begins to come off. The water so-removed is not easily regained by the coating, and until the silicate is re-hydrated, it is insoluble in water. The more water that is removed, the more difficult the subsequent hydration, until the coating is not even re-hydrated (and therefore remains insoluble) in steam at atmospheric pressure. In such cases, the coating resembles the originally fused silicate before its solution in water to produce the commercial liquid (which is made by hydrating the silicate with high pressure steam). Such an insoluble material is produced only at baking temperatures of around 800° F. or higher. Although the coatings may be rendered satisfactorily insoluble by heat treatments as low as 300° F., it is preferable to employ temperatures of from about 480° F. to 600° F. for times of from 1½ to 10 minutes. However, when an appliance application involves the use of hot water or steam, it may be desirable to prebake the coatings at from about 500° F. to 700° F. for 1½ to 10 minutes and then bake the stainless steel article at temperatures of from about 800° F. to 1250° F. for times of from about 2 minutes to 5 minutes. The latter treatment effects a nearly completely insoluble, substantially transparent coating.

It is obvious that coatings with different solubility characteristics are obtained by different baking schedules. Although it is always desirable to have as insoluble a coating as possible, practical considerations usually force a compromise. Continuous coating lines for strip have ovens which seldom go above 650° F., and the available time-in-oven is quite limited because of minimum practical line speeds and maximum practical oven length. Batch or conveyor ovens for sheet coating can give high temperatures and longer times, but if temperatures much above 650° F. are used, experiments suggest that prebaking for several minutes below this temperature but above 300° F. is necessary to keep the coating transparent. This is probably due to too rapid release of water and subsequent frothing of the film.

Experiments to date with the durability characteristics of coatings in exposure, hot water submersion, humidity and steam tests indicate that for most decorative uses, the ultimate in insolubility is not necessary and practical compromises are not detrimental to coating life. Direct flame impingement should be useful, especially for repair work, since the coating, wet or dry, is not flammable.

The heat transfer rate of the oven or heater is important, since for short times this helps determine the actual temperature reached at the surface. Also, freedom of air

movement over the surface is necessary to carry away water removed from the coating. When greater baking times are possible, these factors are less important than when the time is limited. In the latter case, minimum baking times may vary from one oven to another, even though all have the same nominal temperature.

EXAMPLES

Numerous panels of AISI Types 430 and 302 stainless steel, some of each type of which exhibited an "architectural" surface which constituted a buff surface (from steel sheets passed through rolls which had been shot blasted) and some of each type of which exhibited a dull No. 2 strip finish (known as a pickle finish) were partially silicate coated by immersing the samples half-way into a clear, aqueous solution that was made up of 50 ml. of sodium silicate solution, technical 40-42° Bé., and 50 ml. of clear water. After coating, the panels were all baked at temperatures of about 480° F. for thirty minutes. The resulting silicate coatings were .001" or less in thickness. These samples were all scratched several times by dragging the sharp edge of a rectangular stainless steel panel across the surface of each panel so that the scratch extended from within the coated area of the sample to the non-coated area. The scratches in each instance were visible and readily observed where present on the non-coated surface, but were difficult to observe on the coated areas and could be seen only by close observation or magnification. It was also noted that the non-coated areas of these panels quickly acquired visible fingerprint marks due to handling (particularly the No. 2 finish samples), while the coated part of each sample remained substantially free of such marks. Also, it was noted that smudge or grease marks purposely applied to the surface of the panels could be easily removed from the coated portions by simply wiping with a clean cloth while the uncoated portions retained vestiges of the smudge even after scrubbing with soap and water.

Panels of AISI Type 430 stainless steel which exhibited a "bright" No. 2 strip finish (known to the trade as an "automobile finish") were silicate coated as above. The silicate coated portions of these samples exhibited resistance to fingermarks and smudges and were exceedingly easy to clean, while the uncoated portions exhibited fingermarks whenever touched and were difficult to clean.

The following examples will illustrate specific applications of the present invention with respect to the prevention of heat tinting, but in no way limit or restrict the claims to the embodiments set forth: Stainless steel strip and sheet samples (approximately 3" x 6") were brush cleaned in strong hot, alkaline detergent, rinsed, dried and tested as follows:

(1) Type 430 stainless steel strip samples with a bright annealed surface were dip-coated in 65% sodium silicate solution [65 volume percent of 40-42° Bé. sodium silicate solution in water+.1% by weight of Wetanol (wetting agent)] and baked at 500° F. for 10 minutes.

(2) Type 430 stainless steel strip samples with a bright annealed surface were dip-coated in 50% sodium silicate solution and baked at 500° F. for 10 minutes. The process was repeated to give the necessary coating thickness.

(3) Type 304 stainless steel samples with a #4 sheet finish were dip-coated in 50% sodium silicate solution and baked at 600° F. for 5 minutes.

(4) Type 304 stainless steel samples with a #4 sheet finish were sprayed with a 30% sodium silicate solution and baked at 600° F. for 5 minutes.

The degree of roughness of the surface determines the solution concentration necessary to achieve the necessary coating thickness by any given coating method. This thickness is about .05-.1 mil for most uses. Too thin a coating (less than about .01 mil) gives interference fringe colors and inadequate protection, while excessive thickness (more than about 1 mil) results in loss of coating transparency after baking or in-use heating. On a #4

finish, for example, dip-coating in 50% solution gives a coating thickness of .05-.1 mil which is optimum for that finish. Rough surfaces require greater thickness (up to .001") to ensure covering all the substrate "peaks." The most general solution concentrations (based on 40-42° Bé. sodium silicate solutions as 100%) range from 10% to 75% by volume, with 30% and 50% solutions used most often.

PROPERTIES

(1) Appearance

When properly applied, the coating had very little effect on the appearance of the original metal surface. On some finishes the coated surface appeared somewhat darker and slightly more "bluish" than uncoated material, but the degree of this difference depends on the viewing conditions. On certain highly buffed, very bright surfaces, the coating tended to dull the surface slightly.

(2) Flexibility

The ability of the coated stainless to withstand bending, forming and impact varied with surface and coating thickness. In general, the thicker the coating and the smoother the finish, the less the flexibility. On a "Tampico" brushed finish, the coating could be bent sharply (on a bending brake) 90° without loss of protection. On duller finishes, bending could be more than 90°, but on brighter finishes such as a #4 finish, 90° outside bends suffered loss of heat tint protection, although inside bends even greater than 90° remained protected. Where outside bends on bright finishes must be used, a larger radius was necessary.

(3) Solvent resistance

Immersion of the coated stainless steel samples in such solvents as benzene, petroleum ether, acetone, xylene, alcohol, carbon tetrachloride, methyl-ethyl-ketone and trichloroethylene for a period of five days indicated that organic solvent have no effect on the coatings.

(4) Resistance to water

The coated and baked stainless steel samples were placed under hot running tap water for 10 months with no damage to their coatings or surfaces and other samples were exposed in a 100% relative humidity chamber for 10 months with no effect on the coating. Several samples of the coated Type 304 stainless have been under test in a domestic dishwasher for more than a year. At the rate of about 10 washing cycles per week, these samples have withstood in excess of 500 cycles without any effect.

(5) Protection against heat tint

To be useful for decorative applications, the coating must not only stop the effects of heat but must itself remain substantially invisible and unchanged. The data of Table III below shows the time-temperature range for which the coating fulfills the above needs when applied at optimum thickness (about .05 mil) on Type 304 stainless with a #4 (polished) finish. Protection was afforded for an indefinite period of time (3 weeks here) at temperatures of 750° F. or below. Above this temperature, the allowable time drops rapidly—about 1 day at 900° F. and 1 hour at 1000° F. At temperatures above 1000° F. for any substantial period of time (i.e., longer than a few minutes), the coating is no longer suitable for decorative purposes.

(6) Weldability

The coating itself is an electrical insulator, and if continuous on the surface will prevent spot welding. Once electrical contact is established, the coating does not appear to interfere with welding or with the soundness of the weld. On smooth surfaces, abrasive or chemical removal of coating in the weld area may be necessary prior to welding except where butt welding of uncoated edges is to be done. In any welding operation, the coating will

help to confine heat discoloration to the immediate weld area.

The data shown in Table III below tabulates the test results of various samples having various numbers of coats of sodium silicate applied as described. A 50% solution, which was used, effected a baked coating approximately .05 mil thick. Samples with more than one coating (50% solution) had a total coating which was approximately the number of coats multiplied by .05=mils thickness. The 25% solutions effected a coating approximately .025 mil thick.

Table III

TYPE 304, #4 FINISH

[All samples dip-coated and cured for 15 minutes at 550° F. after each coat]

Code	Percent solution	No. of coats	Treatment	Appearance after test
1.....	25	1	550° F., 3 wks.	Clear
2.....	50	1	550° F., 3 wks.	Do.
3.....	50	2	550° F., 3 wks.	Do.
4.....	50	3	550° F., 3 wks.	Do.
5.....	50	4	550° F., 3 wks.	Do.
6.....	25	1	750° F., 3 wks.	Slightly oxidized.
7.....	50	1	750° F., 3 wks.	Clear.
8.....	50	2	750° F., 3 wks.	Slightly milky.
9.....	50	3	750° F., 3 wks.	Opaque.
10.....	50	4	750° F., 3 wks.	Do.
11.....	25	1	900° F., 1 hr.	Clear.
12.....	50	1	900° F., 1 hr.	Do.
13.....	50	2	900° F., 1 hr.	Slightly opaque.
14.....	50	3	900° F., 1 hr.	Opaque.
15.....	50	4	900° F., 1 hr.	Do.
16.....	25	1	900° F., 1 day.	Clear.
17.....	50	1	900° F., 1 day.	Do.
18.....	50	2	900° F., 1 day.	Opaque.
19.....	50	3	900° F., 1 day.	Do.
20.....	50	4	900° F., 1 day.	Do.
21.....	25	1	900° F., 2 wks.	Oxidized.
22.....	50	1	900° F., 2 wks.	Do.
23.....	50	2	900° F., 2 wks.	Oxidized, opaque.
24.....	50	3	900° F., 2 wks.	Do.
25.....	50	4	900° F., 2 wks.	Do.
26.....	25	1	1000° F., 10 min.	Clear.
27.....	50	1	1000° F., 10 min.	Slightly milky.
28.....	50	2	1000° F., 10 min.	Slightly opaque.
29.....	50	3	1000° F., 10 min.	Opaque.
30.....	50	4	1000° F., 10 min.	Do.
31.....	25	1	1000° F., 1 hr.	Clear.
32.....	50	1	1000° F., 1 hr.	Slightly opaque.
33.....	50	2	1000° F., 1 hr.	Opaque.
34.....	50	3	1000° F., 1 hr.	Do.
35.....	50	4	1000° F., 1 hr.	Do.
36.....	25	1	1000° F., 1 day.	Oxidized.
37.....	50	1	1000° F., 1 day.	Do.
38.....	50	2	1000° F., 1 day.	Slightly oxidized, opaque.
39.....	50	3	1000° F., 1 day.	Do.
40.....	50	4	1000° F., 1 day.	Do.
41.....	25	1	1000° F., 2 wks.	Oxidized.
42.....	50	1	1000° F., 2 wks.	Oxidized, opaque.
43.....	50	2	1000° F., 2 wks.	Do.
44.....	50	3	1000° F., 2 wks.	Do.
45.....	50	4	1000° F., 2 wks.	Do.
46.....	25	1	1200° F., 10 min.	Slightly oxidized.
47.....	50	1	1200° F., 10 min.	Very slightly oxidized, slightly opaque.
48.....	50	2	1200° F., 10 min.	Slightly oxidized, opaque.
49.....	50	3	1200° F., 10 min.	Do.
50.....	50	4	1200° F., 10 min.	Do.

Although several examples of this invention have been shown and described, various adaptations and modifications may be made without departing from the scope of the appended claims.

I claim:

1. In a stainless steel member having a decorative surface thereof disposed to be visible in use, the combination comprising, a coating disposed in adhering relation on said surface, said coating comprising a substantially invisible and water insoluble baked-on alkali metal silicate layer having a thickness not exceeding about 0.001 inch.

2. The combination of claim 1 wherein said alkali metal silicate is sodium silicate.

3. The combination of claim 1 wherein said member is a flat rolled stainless steel product having a predetermined light reflectivity surface finish on the surface that is to be visible.

4. The combination of claim 3 wherein said member is an architectural member and said surface finish is not greater in light reflectivity than a No. 4 sheet finish, said coated surface being characterized by its resistance to adverse appearance of mechanical scratches and marks and resistance to fingermarks or smear marks.

5. The combination of claim 3 wherein said surface finish is greater in light reflectivity than a No. 4 sheet finish and said coated surface is resistant to heat tint when subjected to temperatures of from about 300° to 1000° F.

6. In a stainless steel member having a decorative surface thereof disposed to be visible in use, the combination comprising, a coating comprising an alkali metal silicate baked at a temperature from about 300° F. to about 1250° F. in adhering relation on said surface, said coating having a thickness not exceeding about 0.001 inch and being substantially invisible on said visible surface.

7. The combination of claim 6 wherein said alkali metal silicate is sodium silicate.

8. In a stainless steel member having a decorative surface thereof disposed to be visible in use, the combination comprising, a coating comprising an alkali metal silicate baked at a temperature from about 480° F. to about 600° F. in adhering relation on said surface, said coating having a thickness not exceeding about 0.001 inch and being substantially invisible on said visible surface.

9. The combination of claim 8 wherein said alkali metal silicate is sodium silicate.

10. A stainless steel article having a decorative surface thereof disposed to be visible in use comprising flat rolled and formed stainless steel that exhibits a surface finish that has greater light reflectivity than a No. 4 sheet finish and which has a continuous thin substantially invisible water insoluble alkali metal silicate coating on said surface that is approximately .001" maximum in thickness, said article being characterized by its resistance to heat tint when subjected to temperatures of from about 300° F. to 1000° F.

11. A stainless steel architectural member comprising flat rolled stainless steel having a roughened surface that is not greater in light reflectivity than a No. 4 sheet finish and said surface having a substantially invisible and water insoluble alkali metal silicate coating thereon, said silicate coating not exceeding about .001" in thickness, said architectural member being characterized by its resistance to the adverse appearance of mechanical scratches and marks and resistance to fingermarks or smear marks.

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