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HEAT-RELEASING ANTI-ICING MEANS

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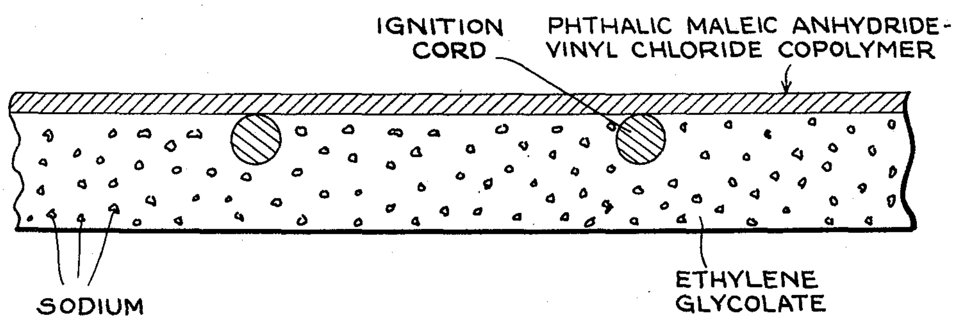

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HEAT-RELEASING ANTI-ICING MEANS

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1 Claim. (Cl. 126—263)

This invention relates to chemical agents and coatings made therefrom which are useful in preventing ice formation on surfaces subjected thereto, particularly air-borne surfaces. More specifically it relates to sodium dispersions which react with impinging water in an exothermic reaction to yield heat, first to prevent ice from forming and second, to melt the ice that has already formed. The instant invention also relates to such dispersions coated with a water-impervious organic film by the use of which de-icing action may be initiated and controlled.

An object of the instant invention, therefore, is to provide coating materials which react with cold water to release heat and thereby prevent such cold water from forming ice on a surface sought to be protected therefrom.

Another object of the instant invention is to provide anti-icing coating means which not only generate heat upon reaction with water but additionally give rise to freezing point depressant materials.

Still another object of the instant invention is to provide an anti-icing compound composed of a sodium dispersion in a non-polar matrix.

A further object of the instant invention is to provide a coated sodium dispersion de-icing material which is activated when needed and which operates automatically subsequent to activation.

A specific object of the instant invention is to provide anti-icing coatings composed of a sodium dispersion in polymerized polyethylene glycolate.

Other objects, features and advantages of the instant invention will be obvious to those skilled in the particular art from the following detailed disclosure hereof.

The one drawing appended hereto and made a part hereof discloses that embodiment of the present invention wherein an organic film is superimposed upon the ice removal coating herein disclosed.

The heat releasing nature of the instant invention is exemplified by the following reaction:

$$Na+H_2O \rightarrow NaOH+\tfrac{1}{2}H_2+44\text{ kcal./mole}$$

indicated, the thermal energy involved in this reaction is considerable and is the primary ice preventative action hereof. Additionally however, the caustic formed in the reaction acts as a freezing point depressant; thus a sodium dispersion operates in a dual capacity to yield the desirable result of our invention.

Industrial sodium dispersions which are available at the present time use non-polar matrices such as kerosene, mineral oil and hexane. Such dispersions are useful for various purposes when the sodium is reacted with non-polar materials into which the matrix dissolves. When these dispersions are brought into contact with water, only the superficially accessible sodium particles react, after which the reaction stops. If the proportion of

sodium is increased in such a way that the reaction proceeds initially at a measurable rate, the heat evolution becomes considerable and the matrix melts, thus allowing more sodium to react with the water. Within a short time the reaction gets out of control and the hydrogen, sodium, and matrix ignite. In view of this, such dispersions cannot be used in the instant invention. We have found that sodium dispersions in a polar, water-soluble matrix are useful de-icing agents. For example, when sodium is dispersed in high molecular weight polymerized polyethylene glycols the desirable results are achieved. When sodium is first dispersed in such glycols it reacts with the hydroxyl groups thereof to yield sodium glycolate as follows:

$$2Na+HOCH_2(CH_2—O—CH_2)_xCH_2OH \rightarrow NaOCH_2(CH_2—O—CH_2)_xCH_2Na+H_2$$

After this reaction is completed an excess of sodium remains in the metallic state i.e., the sodium is dispersed in a matrix of polymerized ethylene glycolate. The proportion of chemically combined sodium is very small; for example, polyethylene glycol having a molecular weight of 6000 contains only 0.8% of combined sodium.

The instant dispersions are easily prepared by introducing small pieces of sodium into molten polyethylene glycol under an atmosphere of nitrogen. The temperature of the melt is maintained above the fusion point of the sodium (208° F.). At first hydrogen is evolved as the glycolate is formed and after this initial stage no reaction occurs and the sodium may be added more rapidly. In some cases the producer may find it desirable to use a high shear stirrer in the manufacturing process.

These dispersions are hard, yellow or brownish solids at room temperature. They may be handled and stored safely in dry air; however, they react upon water with considerable evolution of heat.

A typical composition of dispersion used in our test is as follows:

	Percent
Sodium	12
Polyethylene glycol (600 mol. wt.)	62
Polyethylene glycols (20,000 mol. wt.)	26

We found that dispersions in mixtures of two glycols are less brittle than dispersions in a single compound.

In the instant dispersions the sodium is divided into particles having diameters between 5 and 50 microns. Since each particle is coated with an inert matrix the dispersions does not oxidize in air. When a particle of sodium combines with water the heat of the reaction is dispersed throughout the inert matrix and the temperature does not rise as drastically as when the bulk metal is used. The rate of the reaction is controlled by the concentration of the metal in the dispersion and the rate of water impingement thereon.

The instant dispersions are extremely effective in keeping the leading edge of a surface subjected to icing free of such ice even under the most adverse conditions. For example, we brushed the above composition in the form of a molten, viscous dispersion onto a test panel as a ¼ to ⅜ inch thick strip tapering from the leading edge. The run off of even the narrowest of strips was found to protect our entire mockup.

The following icing wind tunnel data illustrates the usefulness of the instant dispersions in preventing the formation of ice. The various compositions were brushed onto an aluminum panel of a 36-inch chord mockup and then subjected to the severe operating conditions indicated.

ICING TEST DATA

Sodium dispersion in polyethylene glycol

Sample Number	Mol. Wt. of glycol	Percent Sodium	Temp., °F.	Liquid H_2O Content, g./m.³	Air Velocity, ft./sec.	Duration, Minutes	Coating on Leading Edge	
							Thickness, Inches	All consumed, minutes
1	1,540	15	3	0.5	426	10	1/3	10
2	1,540	15	18	1.5	440	10	1/2	10
3	1,540	15	10	0.5	430	10	1/3	10
4	6,000 (20%) 20,000 (80%)	13.5	9	0.5–1.5	445	14	3/16	7
5	6,000 (30%) 20,000 (70%)	12	15	0.8	208	15	3/16	6
6	6,000 (30%) 20,000 (70%)	12	25	0.5	230	14	1/4	11

Note: In no case did ice form. There was liquid run off.

The above dispersions are continuously effective for as long as the protective layer remains available. There are occasions however, when it is desirable to keep the de-icing system in reserve until the need for its usage arises and it is to this latter problem that the remainder of this disclosure is directed. We think it will be obvious to those skilled in this particular art that by such a method the operator of an aircraft may wait until the most severe icing conditions have been met to activate the de-icing system rather than have the system commence operation as soon as the plane encounters suspended water particles in the air.

We have developed a means whereby our de-icers are activated at will after the ice has been allowed to form. The principle of these coatings and procedures is based upon the property of certain organic films to be insoluble in water of neutral pH but soluble in strongly basic solutions. Thus if the sodium dispersion layer disclosed above is coated with such a film the impinging water does not react with the sodium, and ice will build up on the insoluble film. If a small area of the organic film is destroyed the ice begins to react with the sodium to produce a caustic solution. The latter in turn will dissolve the film in the vicinity of the initially destroyed area and within a short period the interferring film will have disappeared and the ice will rest upon a liquid film of caustic. The ice will then be easily swept away by the force of the airstream or will melt from the heat evolved by the water reacting upon sodium.

The instant materials are prepared by coating a thin layer of a sodium dispersion in polyethylene glycolate with a film of phthalic maleic anhydride-vinyl chloride copolymer. Such copolymer is soluble in acetone and may be deposited in a thin film by dipping the sodium dispersion into the acetone solution.

Activation of the anti-icing composition is effected by setting of a thin ignition cord imbedded in the dispersion along the leading edge of the surface to be protected. We used Thermalite Cord, Type A, as manufactured by the Ensign Bickford Company to operate the instant system. Panels were tested by permitting 1 to 2 inches of ice to accumulate before lighting the fuse. The ice cap came off within 3 or 4 seconds after the fuse was lighted.

Such aforedescribed embodiment of our invention is illustrated in the sole figure appended hereto.

It will be understood that modifications and variations may be effected without departing from the spirit and scope of the instant invention.

We claim as our invention:

An anti-icing film for use upon surfaces subject to ice deposition consisting essentially in combination of: a layer of a sodium dispersion in polymerized ethylene glycolate, said dispersion being formed upon reaction of the following constituents:

	Percent
Sodium particles, 5–50 microns sizes	12–15
Polyethylene glycol, 6,000–20,000 molecular weight	85–88

whereby a portion of said sodium reacts with said polyethylene glycol to form polyethylene glycolate leaving the remainder of said sodium in the unreacted metallic state dispersed in said glycolate, a layer of phthalic maleic anhydride-vinyl chloride copolymer coating one surface of said sodium dispersion layer, and ignition cord means embedded in said sodium dispersion layer which upon ignition is capable of rupturing said copolymer layer superimposed thereover.

References Cited in the file of this patent

UNITED STATES PATENTS

2,143,482	Herrmann et al.	Jan. 10, 1939
2,258,184	Keenog	Oct. 7, 1941
2,346,891	Adlington	Apr. 18, 1944
2,431,885	Palmer	Dec. 2, 1947
2,566,923	Burghart	Sept. 4, 1951
2,716,068	Fain et al.	Aug. 23, 1955
2,731,353	Fain et al.	Jan. 17, 1956

FOREIGN PATENTS

744,385	Great Britain	Feb. 8, 1956