PREPARATION AND ANALYSIS OF HIGH TEMPERATURE PROTECTIVE COATING

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Abstract - The development of jet aero-engines and rapid advancements in aeronautical, aerospace, automobile and advanced power generation industries created demands for high temperature structural materials. In modern aero-engines, base alloys (substrate) have been designed primarily for light weight and high temperature strength and these advanced materials may not provide optimal corrosion or oxidation resistance. In such cases, the only option is to rely on effective surface coatings to prevent or minimize sulphidation and corrosion problems. A novel coating of high performance polymeric material is a need of today. These polymeric materials have superior mechanical, thermal and anticorrosive characteristics ideally suitable for adverse environmental conditions. High temperature protective coatings are specially manufactured to withstand extremely high temperatures for longer durations. These paints do not degrade quickly compared to their normal counterparts and are resistant to high temperature, heat and thermal variations. Heat-resistant paints have various industrial applications, including pipes, petrochemical tanks, silencers, boilers, chimney stacks, and furnaces and their piping structure. These paints are different from fire-retarded coatings, which are usually used to protect metal surfaces. These coatings are not designed to extinguish fire. The coating only reduces the chances of fire. High temperature protective coating are in-tumescent and swells up and increases in volume when subjected to high temperatures, the swell up coating produces a shielding substance which discourages the heat conduction and this leads to control the fire.

***Key Words*: solvent losses, coating, Pleasant appearance, light intensity, low-cost.**

1.INTRODUCTION

Coatings are usually applied as multi-layered systems that are composed of primer and topcoat. However, in some cases – for example automotive coating systems, this may vary from four to six layers. Each coating layer is applied to perform certain specific functions, though its activities are influenced by the other layers in the system. The interactions among different layers and the interfacial phenomenon play an important role in the overall performance of the multi-coat systems. Different properties of coatings are typically associated with specific parts of a coating system. Coatings may be applied as liquids, gases or solids. A coating is a covering that is applied to the surface of an object, usually referred to as the substrate. Process of coating involves application of thin film of functional material to a substrate. The functional material may be metallic or non-metallic; organic or inorganic; solid, liquid or gas.In many cases coatings are applied to improve surface properties of the substrate, such as appearance, adhesion, wet ability, corrosion resistance, wear resistance, heat resistant, and scratch resistance. The principal mechanisms by which binders form films are reaction with oxygen from the air (oxidation), evaporation of the solvent from the vehicle (solvent evaporation), or chemical cross linking (polymerization). The coating film attained by these mechanisms can be either thermoplastic or thermosetting. Thermoplastic materials deform and soften on exposure to heat. Thermoset materials do not deform and remain hard upon heat exposure. Protective coatings are used in oil and gas storage, transmission and distribution network to prevent steel structures against corrosion. Steel structures like trains, ships, automobiles, airplanes, underground buried gas pipelines, fuel storage tanks etc require the use of protective coatings. The importance of a coating can be judged from the fact that it can hardly be ignored in any corrosion protective scheme. Protective coatings are unique specialty products which represent the most widely used method of corrosion control. They are used to give long term protection under different corrosive conditions. The function of a protective coating or lining is to separate two highly reactive materials; i.e. to prevent corrosive environment species from contacting the reactive underlying steel structure. This is to say that a coating acts as a barrier to prevent either chemical compounds or corrosion currents from contacting the substrate.

1 Objectives of project

1. To develop the blend of Epoxy - Silicone resin.
2. To characterised the physical and chemical properties of paint.
3. To analyse the High temperature protective coating property with different pigments and solvent.
4. To study the % loss of heat after applying paint of different coat thickness.

1.2 Epoxy Resin Synthesis Epoxy resins are compounds containing more than one epoxide group per molecule on average. Commercial epoxy resins contain aliphatic, cycloaliphatic, or aromatic backbones. They are prepared from either epichlorohydrin or by direct epoxidation of olefins with per acids. The most important intermediate for epoxy resins is the diglycidyl ether of bisphenol A (DGEBA), which is synthesized from bisphenol A and excess epichlohydrin. Commercial liquid epoxy resins are prepared using different molar ratios of epichlohydrin to bisphenol A to afford different molecular weight products. High molecular weight solid epoxy resins with n values ranging from 2 to 30 are prepared by two process. Lower molecular weight solid resins with n values up to 3.7 are prepared directly from epichlorohydrin, bisphenol A and a stoichiometric amount of NaOH (taffy process). Higher molecular weight solid resins are prepared by chain extension reaction of liquid epoxy resin (crude DGEBA) with bisphenol A using basic inorganic reagents such as NaOH or Na2CO3 as catalysts (advancement or fusion process). Most epoxy resins are produced from a reaction between epichlorohydrin and bisphenol. When epichlorohydrin and bisphenol-A mix together to form a covalent bond, the resulting polymer resin is Epoxy resin Epoxy adhesion is due to the strong polar bond the epoxy makes with the surface. Also used for protective coatings. Epoxy resins are polymeric or semi-polymeric materials, and as such rarely exist as pure substances, since variable chain length results from the polymerization reaction used to produce them. High purity grades can be produced for certain applications, e.g. using a distillation purification process. One downside of high purity liquid grades is their tendency to form crystalline solids due to their highly regular structure, which require melting to enable processing. An important criterion for epoxy resins is the epoxide content. This is commonly expressed as the epoxide number, which is the number of epoxide equivalents in 1 kg of resin (Eq./kg), or as the equivalent weight, which is the weight in grams of resin containing 1 mole equivalent of epoxide (g/mol).

Equivalent weight (g/mol) = 1000 / epoxide number (Eq./kg)

1.3 Properties of Epoxy resin

1. Good Chemical Resistant
2. Good Physical Properties
3. Good adhesion to metals
4. Low Thermal conductivity
5. High Mechanical Properties: large cohesive force

Good Adhesive Performance: renumber of polar group
6. Excellent stability: without impurities(base and salt)Flexibility and diversity in Designing
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2. Silicone Resin :

Silicones are organic silicon compounds known as polyorganosiloxanes. This product group can be defined simply as follows:

1.They are polymers

2.Silicone is directly bonded to carbon

3.There is at least one oxygen atom connected to the silicon

The silicones occupy a hybrid position between inorganic and organic compounds, specifically between silicates and organic polymers. The siloxane link resins synthesis (Si-O-Si), also found in silicates, is responsible for the "inorganic" character. The organic properties arise from the direct link between silicon and carbon.

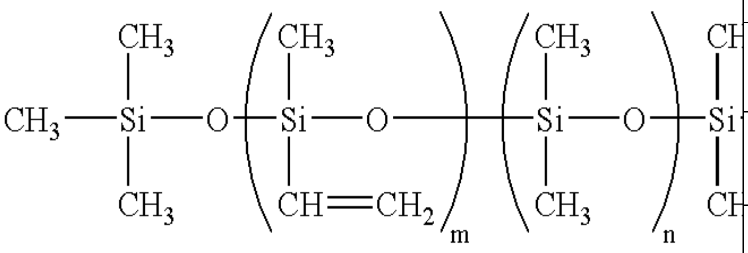

Fig (1) : Silicone Resin Structure

3 Solvents

Solvents are one of main ingredients of the paints and are used to obtain proper viscosity. Sometimes the prepared paint is made without all the liquated. These coatings are then thinned before use to desired consistency by adding a thinner diluents On the basis of their chemical characteristics, solvents divided in separate groups, e.g. crude oil based aliphatic and aromatic solvents, oxygen-containing ketone alcohols, esters and water. A solvent is typically selected based on its ability to dissolve binder components (resins), and its evaporation rate. Its ability to dissolve binder components is often referred to as solvent power. Combinations of different solvents are often found in paint formulations. The most widely used solvents are toluene, xylene, MEK and MIBK.

4. MATERIALS AND METHODOLOGY

Materials :

The main constituents of paints are Resin, Pigments, Solvents and Additives. The raw materials for the preparation and analysis of coating are

4.1Resins

1.Epoxy Resin
2.Silicone Resin
3.Polyamide Resin as Hardener

Table 1- Physical and Chemical Properties of Resins

Epoxy Resin	Silicone Resin	Hardener
Atul Limited	Dow Corning	Atul Limited
Clear viscous liquid	Liquid	Clear liquid
Lapox® L - 12	Xiameter(R) Rsn-0808 Resin	Lapox® K-6
Density1.1 – 1.2g/cm3	Specific Gravity @ 25°C: 1.006	Density 25ºC 0.95 – 1.1 g/cm3
Viscosity9000 – 12000mPa.s	Viscosity at 25°C - 80 to 200 mPa.s	Viscosity 20ºC 5 – 10 mPa.s

Table 2- Physical and Chemical Properties of Pigments

Titanium Dioxide	Carbon Black	Aluminium Paste
Vijay Paints	Abbey Chemicals	Metal powder company ltd
White Solid. (Powdered solid.)	Powder, dust- Black	Semi-Solid
Melting Point: 1855°C	MELTING POINT (°C) >3000	Melting Temperature 660°C
Boiling Point: 2750°C	BOILING POINT (°C) >3000	Boiling Temperature -2467°C
Specific Gravity: 4.26	DENSITY 1.7-1.9g/cm3	Density 1.4-1.6 gm/cm^3

Table 3- Physical and Chemical Properties of Solvents

Xylene	MIBK	Acetone
Lobachem Company	Lobachem Company	Lobachem Company
Liquid. Colour: Colourless. Clear	Liquid.	Liquid. Colour - Colourless
Vapour Pressure: 0.9 kPa (@ 20°C)	Vapour Pressure: 15.7 mm of Hg	Vapour Pressure 0.4 mmHg
Specific Gravity: 0.864 (Water = 1)	Specific Gravity: 0.802 (Water = 1)	Specific Gravity (H2O = 1) 0.9005 - 0.9040 20 °C/
Molecular Weight: 106.17 g/mole	Molecular Weight: 100.16 g/mole	Molecular Weight 118.2 g/mol

5 Batch Preparation

Batch A - Epoxy- silicone resin 50 %-50%

Batch B - Epoxy- silicone resin 55 %-45%

Batch C - Epoxy- silicone resin 75%-25%

Batch D - Epoxy- silicone resin 65%-35%

5. 1BATCH PREPARATION

Batch A - Epoxy- silicone resin 50 %-50%

❖ **Preparation of Panels**

5.2 Preparation of Panels

The panels which are used for a testing purpose have

Material	Quantity	Stirring time
1) Epoxy Resin	50ml	10 min stirring
2)MIBK,Acetone, Xylene	5ml (1:1:1)	
3)Silicon	50ml	10-15 min stirring
4) Xylene	5ml	
5)Pigment (Wt basis) Epoxy Resin + Silicon Resin	11.1 gm/15.5 gm	10 min stirring
6)PolyamideResin (According to Stoichiometric calculation of curing agent)	27.84ml	10 min stirring

been degreased with trichloroethylene and then san blasted to remove rust and mill scale from the surface of the iron.

Apply the paint by brush on test panel. The painted panels are allowed to cure at ambient condition. The coating thickness (average four locations on the test specimen) is measure. Check the heat resistant properties at different thickness

Application of Paint: Manually by brush.

6. EXPERIMENTAL RESULTS

1 Testing of Paint

1) Density Measurement :
2) Viscosity:
3) Pot life
4) Drying Time
5) Curing Time
6) Surface to Touch Time
7) Complete Dry time

1 Density Measurement :

The density of the liquid paint is determined by means of weight per volume of density bottle. The bottle has known volume. The difference between in weight of bottle filled with paint and empty bottle gives the weight of the known volume of paint.

2.Viscosity:

Viscosity is described as the internal resistance of a fluid to flow and may be considered as a measure of fluid friction. It is generally considered as flow behaviour or resistance to pouring. Viscosity is an important parameter in the paint industry processing stage. The test instruments consist of cup with an orifice at the bottom. Both cup and orifice are controlled volume diameter and length. The ford cup is widely used for viscosity determination.

1. Adjust the sample to measuring temperature, usually 25°C – 35°C
2. Make sure that the cup is clean and place it into its support.
3. Close the orifice of the cup with a finger and fill to overflowing with the conditioned sample. Note that the finger can be removed from the orifice when the cover plate is in place.
4. Start a timer as the cover plate is removed from the cup.
5. Determine the temperature of the efflux stream. If there is a significant change during the test it should be repeated.
6. Stop the timer at the first break of the efflux stream, one or two inches below the orifice.

7. The time required for the paint to flow completely through the orifice is measured.

Photo 1– Ford Viscosity Cup

3. Pot life

Pot life is the time from combining the components of the paint to the point at which the mixed paint is no longer useable. It is period of time during which paint remains useful after its original package has been opened or after a catalyst or other additive has been incorporated. It is sometimes also called the working time or useable life.

4.Drying Time

The paint drying process corresponds to the evaporation of all solvents and diluents added to the paint in order to make it liquid or reduce its viscosity. It is the time required for paint to dry is determined by spreading a sample on glass plate placed vertically in a well ventilated room and making a quantitative measurement of time required to dry as determined by finger touch of the surface

5.Curing Time:

The time in which an resin undergoes a molecular reaction and becomes a solid. Curing may require light, heat, or the passage of a specific amount of time.

7. Physical and Mechanical Properties of Painted

1panels
2Dry film Thickness.
3Adhesion Test :
4Water Absorption test - ASTM D570
5Impact Resistant Test
6Scratch Hardness

1. Dry Film Thickness for all batches samples

Test	Observation
Dry Film Thickness	Ranges from 80 to 100 micron

2.Adhesion test for Batch B - Epoxy- silicone resin 55 % - 45%

Adhesion test	There is no detachment of film of coated panels

2.Water Absorption test - ASTM D570

Water absorption is used to determine the amount of water absorbed under specified conditions. For the water absorption test, the coated panels are dried in an oven for a specified time and temperature and then it is cooled at room temperature. Immediately upon cooling the coated panels are weighed and these panels are immersed in a water bath. After 24 hours, it is taken out and the excess water is removed by the filter paper and then weighed. This procedure is repeated till the identical values are obtained. From the amount of water absorbed the percentage of water uptake is calculated.

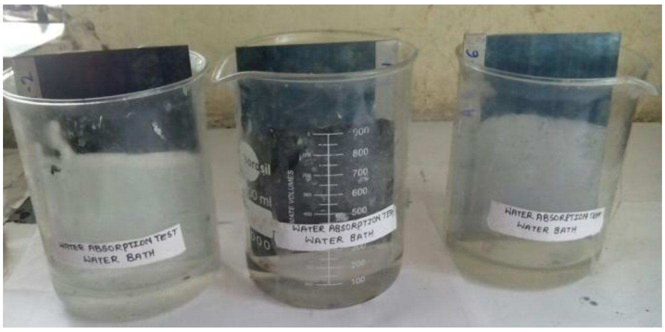

Photo 2 Water Absorption test

3.Impact Resistant Test :

This was measured by falling weight method. In this test a tub of weight is allowed to fall over the painted specimen from a known height. The specimen is then examined for any damage to the film.The organic coatings under test are applied to four or more suitable thin metal panels. After the coatings have cured, a standard weight is dropped a distance to strike an indenter that deforms the coating and the substrate. The indentation can be either an intrusion or an extrusion. By gradually increasing the distance the weight drops, generally 1 inch (25 mm) at a time, the point at which failure usually occurs can be determined

Photo 3 – Impact Resistant tester

4. Scratch Hardness

In general, hardness measures the resistance of materials to permanent or plastic deformation. When testing coatings, scratch hardness refers to the force necessary to cut through the film to the substrate. Placed the painted panels are kept under the test to a scratching motion of the brass rod, using a force of ½ kg and 1 kg. Panels coating are considered to be soft, if during the scratching process, a groove is made in them without deposition of metal from the brass rod, or if separate the coating film from the surface of the painted panels. The panels were loaded with different weight until a clear scratch showing the bare metal surface was seen.

Photo 4– Scratch Hardness tester

8. CONCLUSION

1.The "hybrid" chemistry achieved by combining an aliphatic epoxy with a silicone resin allows the formation of a silicone-epoxy resin that performs better than an organic or inorganic polymer alone. This allows for a durable binder for the protective coatings industry.
2.The development of this paint reduces the cost.
3.The Paint can be used for the corrosion protection and decorative purpose.
4.It offers improved Chemical properties in immersion test, solvent resistant test, and water resistant test.

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