APPLIED GRAPHENE MATERIALS

TECHNICAL APPLICATION NOTE

Anti-Corrosion Primers: Part 3

Active Non-metallic Anti-Corrosion Additives

Contents

1. Introduction

Applied Graphene Materials UK Ltd (AGM) manufacture graphene nanoplatelets using a proprietary and patented process developed at Durham University in the UK.

Applied Graphene Materials UK Ltd have developed significant in-house knowledge on the behaviour and performance of graphene modified coating formulations. These guidance notes are designed to provide formulation insights to assist development scientists achieve a technical appreciation of this novel technology.

The addition of graphene alone by a **G*enable*® ** dispersion into coating formulations has demonstrated excellent barrier properties leading to a reduction in water vapour transmission rates (WVTR). This reduction in WVTR significantly enhances the anti-corrosion performance of an epoxy coating. (This improvement is discussed in more detail in AGM's Technical Guidance note on Anti-Corrosion Primers: Part 1).

This technical application note describes in further detail how the addition of a **G*enable*® 3000** series dispersion to a base paint can lead to significant improvements in the anti-corrosion performance of a primer and the potential benefits this can provide for a customer.

2. Inhibitors

An ideal inhibitive coating should form a barrier against water and detrimental ions whilst simultaneously releasing a sufficient quantity of inhibitor. These two requirements are antagonistic in principle and require a balance in the pigment with a dependency on the barrier properties of the coating.

In previous work (Technical Application: Anti-Corrosion Primers: Part 2) a range of inhibitors were used. Further developments from this work have led to a novel anti-corrosive pigment providing excellent anti-corrosion performance.

Inhibitive coatings are generally applied in the form of primers and are used where the substrate is exposed to atmospheric corrosion and not where the substrate is immersed in water or soil.

The selection of an active pigment has been coming under ever increasing environmental and regulatory pressures leading manufacturers to remove and/or reduce the amount of metal salts used in coating systems.

3. Anti-Corrosion Evaluation of Graphene and Non-Metallic Pigment Dispersion

G*enable*® 3001 (an activated graphene modified pigment dispersion) has been formulated into epoxy based primer system for typical C3 category performance based on ISO 12944. It has been evaluated using the output from the tests listed below.

Testing carried out

Accelerated exposure:	Salt Spray testing	ASTM G-85-94 Prohesion
	Paints and Varnishes: Evaluation of Degradation	ISO 4628-2, 3, 8:2012
Mechanical Testing:	Flexibility	ISO 6860: 6860
	Impact	ISO 6272-2:2004
	Abrasion	ISO 7784
	Adhesion	ISO 4624

3.1. Primer Starting Point Fomulation Based on G*enable*® 3001

The primer was made up using the formulation below resulting in an 87% stoichiometry. It was cured over seven days at ambient room temperature.

Part A: Epoxy Base

	Item	Raw material name	Control (Base Paint plus 8% Zinc Phosphate	Base Paint plus G*enable*® 3001
	Charge items 1,2,3 ,4 and 5 and mix at high speed (2000 rpm) for 10 minutes		%wt	%wt
Charged	1	Epoxy Resin (EEW= 250g/eq.)	11.34	11.34
Charged	2	Cymel U-216 resin	0.25	0.25
Charged	3	Anti-terra U	0.41	0.41
Charged	4	Xylene	7.84	7.84
Charged	5	Tixogel MP	0.37	0.37
	Check Gel is homogenous and free of bits. Continue mixing if not.			
	Add items 6 to 9. Mix at high speed (2000 rpm) for 15 minutes. Check grind <25 microns and add items 6.			
Grind	6	Butanol	2.02	2.02
Grind	7	Titanium dioxide	11.18	11.18
Grind	8	Zinc Phosphate	8	0
Grind	9	Blanc Fixe	37.47	43.47
	Add items 11, 12 & 13. Mix at medium speed (1000rpm) for 15 minutes.			
Let Down	10	**G*enable*® 3001** dispersion addition	0	11
Let Down	11	Epoxy Resin (EEW=250g/eq.)	14.28	4.27
Let Down	12	Xylene	7.84	7.84

Part B: Hardener

The epoxy to hardener mix ratio can be calculated for an appropriate hardener based upon the EEW and AHEW values. In this case an aliphatic amine hardener was used (e.g. Ancamine 2324)

pvc	35	37
VOC (g/l)	320	309

3.2. Manufacturing Guidelines for Anti-Corrosion Coating

It is recommended that **G*enable*®** dispersions should be added at the let-down stage of the manufacturing process.

Impact on PVC: For guidance please contact Business Development on the included contact details.

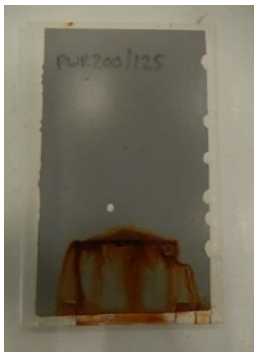
3.3. Test Panel Preparation

Substrate	Cold Rolled Steel
Dimensions	152mm by 101mm
Preparation	Grit blasting to SA2-1/2 rolled by degreasing with acetone
Grit	Irregularly shaped chrome/nickel shot
Application	Spray application (gravity-fed gun 1.2 mm tip)
Coating Thickness	DFT 60-75 µm
Curing	7 Days at Room Temperature

3.4. Salt Spray Testing ASTM G-85-94 Prohesion

Primer Control (with 8% Zinc Phosphate) and **G*enable*® 3001** test panels after 1000, 2000, 3000, 4000 & 5000 Hours of testing to ASTM G-85-94:

	Control (Primer Only) In-house primer + 8% Zinc Phosphate	**In-house primer + G*enable*® 3001**
1000 Hours		

2000 Hours	X	
3000 hours	X	
4000 hours	X	
5000 hours	X	

3.5. Corrosion Rating Test Results (1000, 2000, 3000, 4000 & 5000 Hours)

Anti-Corrosion Pigment (wt/wt)	Prohesion salt spray exposure (hours)	Creep (mm)	Blistering (Qty)	Blister Size	Corrosion	Comments
Zinc Phosphate at 8%	1000	4	1	S3	Ri5	Failed, Corroded across whole face
G*enable®* **3001** at 11%	1000	1	0	S0	Ri2	Good
G*enable®* **3001** at 11%	2000	1	0	S0	Ri2	Good
G*enable®* **3001** at 11%	3000	1	0	S0	Ri2	Good
G*enable®* **3001** at 11%	4000	1	0	S0	Ri2	Good
G*enable®* **3001** at 11%	5000	1	0	S0	Ri2	Good

3.6. Mechanical Testing (7 Day Cure): Adhesion, Flexibility, Impact & Abrasion

Adhesion Testing

	Force (MPa)			Comment
Primary Anti-Corrosive	Rating 1	Rating 2	Average	
Zinc Phosphate at 8%	2	2	2	50% Adhesive failure
G*enable®* **3001** at 11%	1.5	1.8	1.65	Cohesive failure

Conical Mandrel Testing

Primary Anti-Corrosive	Cracking	Elongation
Zinc Phosphate at 8%	4	21
G*enable®* **3001** at 11%	0	<35

Impact Testing

	Cracking begins: Height (cm) 1Kg Weight						
Primary Anti-Corrosive	10	20	30	40	50	60	70
Zinc Phosphate at 8%	X	X					
G*enable®* **3001** at 11%	X						

Taber Abrasion Testing

	100 Cycles, 1Kg Weight, CS-10 Discs			
Primary Anti-Corrosive	Initial Mass (g)	Final Mass (g)	Mass Loss (mg)	Wear Rating
Zinc Phosphate at 8%	68.1826	68.1543	28.3	283
G*enable®* **3001** at 11%	67.3551	67.3154	39.7	397

3.7. Performance

The aim of the work described in this technical application note is to evaluate how active non-metallic anti-corrosion additives could enhance the corrosion protection performance of anti-corrosion primers which in turn could lead to a meaningful extension of coating life.

Given that some of the key anti-corrosion pigments such as chromates and phosphates incorporated into coating systems are coming under increasing environmental pressure to be removed or reduced then viable alternatives will be of interest to the Coatings Industry.

This work has demonstrated that the incorporation of the **G*enable*® 3000** series in a primer can significantly enhance corrosion protection.

4. G*enable*® Storage Stability

AGM graphene is supplied in dispersion format. 12 months stability testing has shown that dispersions in epoxy resins are stable to agglomeration.

	12 months
Syneresis	None
Sedimentation	5mm soft sediment. Easily re-incorporated.
Agglomeration	None

	Dx (10)	**Dx (50)**	**Dx (90)**
Initial	0.83 µm	8.10 µm	31.0 µm
1 month	0.68 µm	7.65 µm	28.8 µm
12 months	0.68 µm	7.27 µm	29.2 µm

Store under ambient conditions for up to 12 months. Dispersion may show slight sedimentation during transportation or on storage. Customer may need to re-agitate by simply mechanically mixing thoroughly with spatula, palette knife or mechanical stirrer before use.