APPLIED GRAPHENE MATERIALS

TECHNICAL APPLICATION NOTE

Waterborne Coatings

Acrylic DTM (Direct-to-Metal)

Contents

1. Introduction

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

AGM have developed significant in-house knowledge on the behaviour and performance of graphene modified coating formulations. These guidance notes are designed to provide information insights to assist development scientists in achieving a technical appreciation of this novel technology.

This technical application note describes in further detail how effective graphene can be in improving the anti-corrosion performance of a waterborne acrylic based coating for Direct-to-Metal (DTM) applications.

2. Direct-to-Metal

Direct-to-Metal coatings, as the name implies, are applied directly onto a metal substrate without the use of a primer beforehand. DTM coatings have an acrylic composition that allows them to successfully adhere to a metal surface when directly applied. Since a primer is not necessary for DTM coatings, they are regularly chosen for jobs when surface preparation is not possible or for applications in which high quality is not the primary concern.

This lack of a need for a primer and reduced levels of surface preparation combined with low levels of VOC emissions make DTM coatings potentially very interesting to operators who are under ever increasing pressures to reduce costs. In this cost sensitive market there is also the desire to improve the anti-corrosion performance of the DTM in harsh humidity conditions.

3. Anti-Corrosion Evaluation

The objective of the work in this technical note was to evaluate and determine if the addition of graphene nanoplatelets can enhance corrosion protection in coating systems to deliver a meaningful extension of life in a waterborne acrylic DTM.

Testing Carried out

Accelerated exposure	Neutral Salt-Spray (Fog) Testing (ASTM B 117—Method 811.1 of Federal **Test** Method 151b): Corrosion Creep Assessment to ISO4628-2-2003 and ISO4628-3-2003

3.1 Typical Graphene Based Formulations for WB Acrylic DTM (Direct-to-Metal)

The evaluation was conducted using the graphene based DTM formulations below.

The DTMs were made up using the respective formulations below. They were cured over a minimum period of 7 days at 23°C.

Table 1: WB Acrylic Preliminary Formulations

		Weight % of G*enable*® 1250 in Formulation				
Item	**Material**	**Supplier**	**Control**	**5% G*enable*® 1250**	**10% G*enable*® 1250**	**20% G*enable*® 1250**
1	De-ionised Water		5.95%	5.65%	5.36%	4.76%
2	Additol® VXW 6208	Allnex	1.81%	1.72%	1.63%	1.45%
3	Additol® VXW 6393	Allnex	0.23%	0.22%	0.21%	0.19%
4	Ti-Pure™ R-706	Chemours	25.96%	24.66%	23.36%	20.77%
5	Acrysol™ RM-2020E	Dow	0.19%	0.18%	0.17%	0.15%
6	Resydrol® AY 6150w/45WA	Allnex	60.26%	57.25%	54.24%	48.21%
7	Ammonia (29%)		0.43%	0.41%	0.39%	0.35%
8	Additol® VXW 6206	Allnex	0.68%	0.64%	0.61%	0.54%
9	Additol® VXW 6503 N	Allnex	0.31%	0.29%	0.27%	0.24%
10	Additol® VXW 4973	Allnex	0.18%	0.17%	0.16%	0.15%
11	Modaflow® AQ-3025	Allnex	0.50%	0.47%	0.45%	0.40%
12	Additol® XL 297	Allnex	0.54%	0.51%	0.48%	0.43%
13	Acrysol® RM-8W	Dow	0.97%	0.92%	0.88%	0.78%
14	Acrysol® RM 2020E	Dow	0.83%	0.79%	0.75%	0.67%
15	Dionised Water		0.17%	0.16%	0.15%	0.14%
16	**G*enable*® 1250**	AGM	0.00%	4.95%	9.90%	19.80%
17	HaloX® Flash-X 150	ICL	1.00%	1.00%	1.00%	1.00%
Total			**100.00%**	**100.00%**	**100.00%**	**100.00%**
pvc			20.08%	25.65%	30.48%	38.46%
VOC (g/l)			10.43	9.91	9.38	8.34

Note: For **G***enable*® **1250** the calculated value of pvc is 69% and the volume solids is 51%. A substantial contributor to these values is the graphene component of the **G***enable*® dispersion. Further a relatively small increase contributes to a large increase in the pvc of the paint formulation. To offset this increase in pvc it will be necessary for the formulator to reduce the bulk filler content of their formulations.

3.2 Manufacturing Guidelines for Anti-Corrosion Coating

3.2.1 Acrylic Based Waterborne Coatings (items referred to in the manufacturing guidelines relate to the items in the above formulation table)

Pigment Paste

Add under agitation with a mechanical mixer. Speed should be adjusted to maintain a consistent vortex.
Add items 1 and 2 and disperse for 5-10 minutes at med speed.
Add items 3 and 4 and disperse for 10 minutes at med-high speed.
Add item 5 and disperse for 20-30 minutes at high speed to obtain a Hegman of 7+.

Let Down

Add under agitation with mechanical mixer. Speed should be adjusted to maintain a consistent vortex.
Add items 6-8 and shear for minimum 10 minutes at high speed.
Add items 9-12 and the pigment paste previously prepared and shear for minimum 10 minutes at low-medium speed.
Add items 13-15 and mix for 10 minutes.
Add items 16 and 17 and mix for 5 minutes at med-high speed.

3.2.2 Acrylic Test Panels

Substrate	Cold Rolled Carbon Steel
Dimensions	150mm by 100mm
Preparation	i) 1Blasted steel (50 to 75 micron blast profile) ii) Q-lab Bonderite steel iii) Q-Lab Abraded steel
Application	150 micron drawdown bar
Coating Thickness	DFT = 50 to 60 microns
Curing	>7 days at 23°C

4 Neutral Salt-Spray (Fog) Testing (ASTM B 117)

4.1 Acrylic Test Panels at 480 Hours

4.1.1 Images at 480 Hours prior to coating being cleaned off for creep assessment

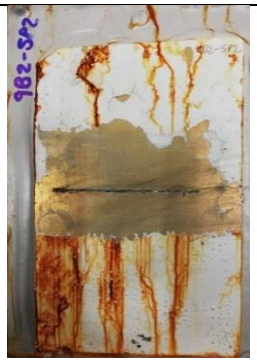
i) DTM Control
ii) Control + 5% **G*enable*® 1200**,
iii) Control + 10% **G*enable*® 1200**
iv) Control + 20% **G*enable*® 1200**
on Q-Lab Abraded Steel, Q-Lab Bonderite Steel and Blasted Steel substrates.

Note: That prior to the coating being removed for creep assessment after 480 hours of neutral salt spray testing that there is a decrease in visible surface corrosion across the series compared to the control panel.

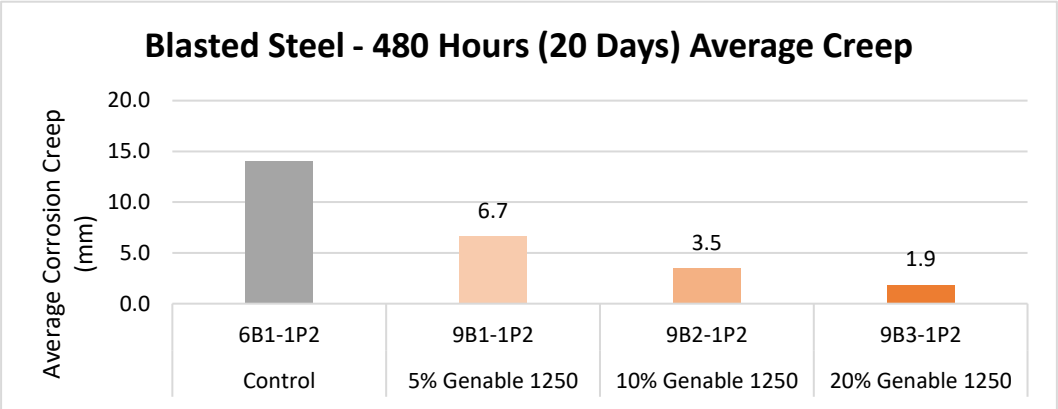
480 Hours	Control	Control + 5% **G*enable*® 1250**	Control + 10% **G*enable*® 1250**	Control + 20% **G*enable*® 1250**
Blasted Steel				
Bonderite				
Abraded Steel				

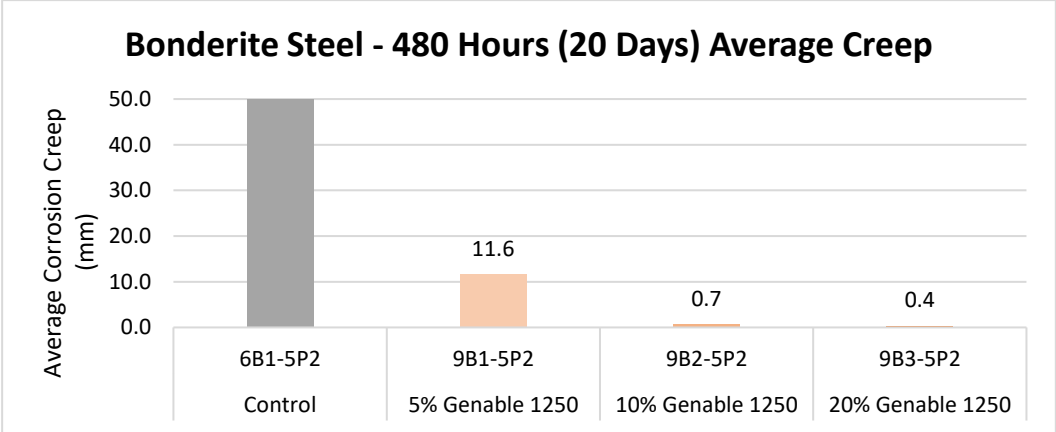
4.1.2 Images at 480 Hours with coating cleaned off for:

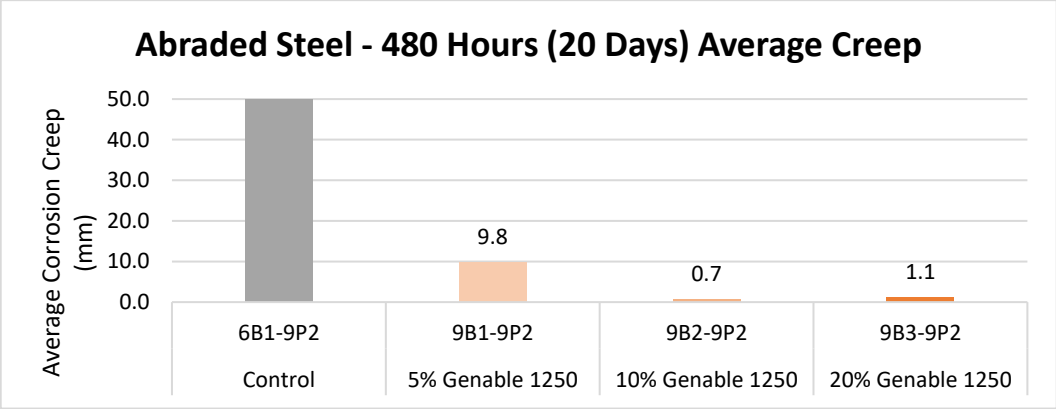
i) DTM Control
ii) Control + 5% **G*enable*® 1200**
iii) Control + 10% **G*enable*® 1200**
iv) Control + 20% **G*enable*® 1200**
on Q-Lab Abraded Steel, Q-Lab Bonderite Steel and Blasted Steel substrates.

480 Hours	Control	Control + 5% **G*enable*® 1250**	Control + 10% **G*enable*® 1250**	Control + 20% **G*enable*® 1250**
Blasted Steel				
Bonderite				
Abraded Steel				

4.1.3 Creep Assessment at 480 Hours







Note: Except for the 480-hour assessment of the coated Blasted Steel control panel all of the other control panels at both 480 hours and 1000 hours testing had substantial levels of corrosion emanating from the scribe and/or a complete failure in terms of corrosion. These panels have been denoted as having an average creep corrosion of 50mm to aid pictorial representation in the graphs above.

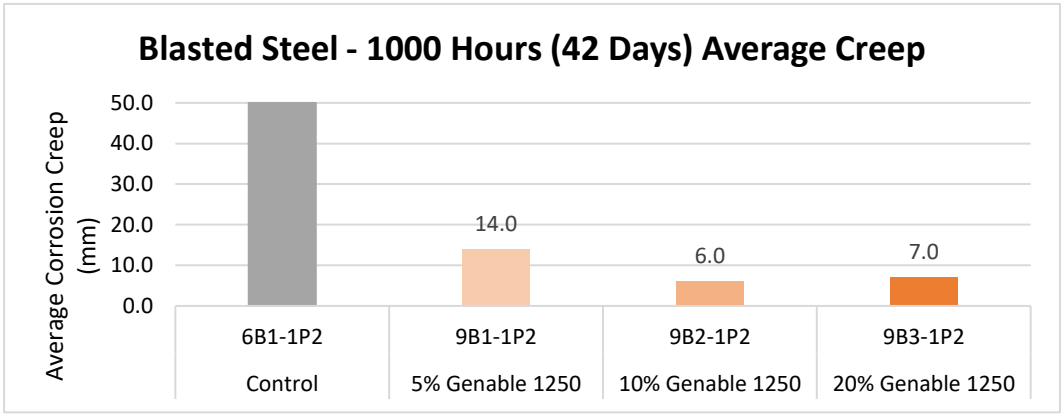
4.2 Acrylic Test Panels Test Panels at 1000 Hours

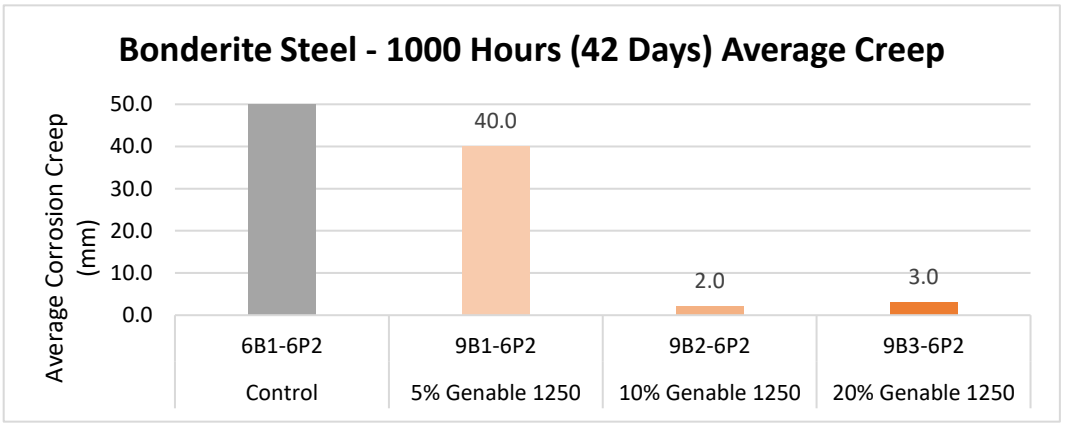
4.2.1 Images at 1000 Hours with coating cleaned off for:

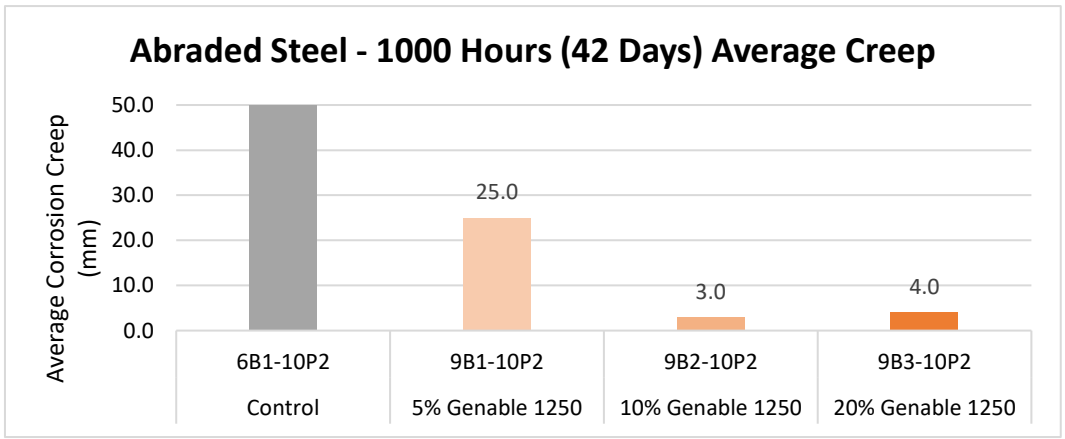
i) DTM Control,
ii) Control + 5% **G*enable*® 1200**,
iii) Control + 10% **G*enable*® 1200**
iv) Control + 20% **G*enable*® 1200**
on Q-Lab Abraded Steel, Bonderite Steel and Blasted Steel substrates.

1000 Hours	Control (DTM)	Control + 5% **G*enable*® 1250**	Control + 10% **G*enable*® 1250**	Control + 20% **G*enable*® 1250**
Blasted Steel				
Bonderite				
Abraded Steel				

4.2.2 Creep Assessment at 1000 Hours







5. Technical Comments

5.1 Assessing whether Graphene can Enhance Anti-Corrosion Performance

The aim of the work described in this technical application note is to evaluate if graphene nanoplatelets could enhance the corrosion protection performance of a DTM coatings which in turn could lead to a meaningful extension of coating life.

In the images shown of the panels in the accelerated exposure tests (Neutral Salt-Spray Fog testing: ASTM B 117) at 480 hours and 1000 hours testing duration respectively; the addition of graphene nanoplatelets to the acrylic formulation has reduced the corrosion observed at the scribe. The reduction in corrosion at the scribe on the test panels is the most pronounced at additions levels of **G*enable*® 1250** at 10% and 20% wt. additions in the tested formulations. It is anticipated that this performance improvement will translate into meaningful extension of coating life for real life applications.

In previous work the addition of graphene by a **G*enable*®** dispersion into solventborne coating formulations has demonstrated excellent barrier properties leading to a reduction in water vapour transmission rates (WVTR) for epoxy coatings alone. It is assumed that a similar mechanism led to the increased anti-corrosion properties observed in waterborne acrylic based coatings.

5.2 Formulating

The **G*enable*® 1200** range has been developed through a large in-house testing programme to demonstrate that graphene could be introduced into a customer's matrix/resin. The starting point formulations outlined above are examples of how the **G*enable*®** dispersions can be used to directly introduce graphene into a water bourne resin formulation. The **G*enable*®** dispersions are typically introduced into the formulations at the let-down stage.

6 G*enable*® Storage Stability

6.1 Storage Stability of Waterborne Products

Waterborne products should be stored in covered, dry conditions and stored in the temperature range 4°C and 35°C.

G*enable*® 1050 has a 3 month shelf life. If settling has occurred during storage then simple mechanical mixing will re-disperse the graphene for use.

G*enable*® 1250 has a 6 month shelf life with minimal sedimentation expected.