Current perspectives on graphene polymer composites as automotive coatings

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Abstract

This short technical review prospect the recent advances of composite coatings reinforced by graphene structures for automotive applications. We summarize the advantages in the use of graphene as a coating additive, mainly focused on the protection mechanisms against corrosion and mechanical damage, the challenges, and perspectives of this innovative class of coatings.
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Background

Steel is one of the most popular engineering materials with widespread industrial applications. Remarkably in automotive field, steel represents more than 50% of the vehicle weight distributed along bodywork, panels, engine, transmission, suspension, and others. [1] Unfortunately, steel undergoes corrosion easily, due to weathering and when it comes in direct contact with oxidative electrolytes. [2] Nowadays, with the emergence of electric vehicles (EV), the concerns with metallic corrosion are even more evident, considering the possibility of leakage of battery acids. Besides that, the use of thinner gauge mild steels, magnesium, and aluminum, required to compensate the heavy weight of the batteries, increase the risks of galvanic corrosion. [3] Thus, corrosion studies on steel parts continues to receive significant interest, and the production of anticorrosion coatings with superior protection efficiency is still a huge topic of advanced research. [4]

Organic coatings act as a passive physical barrier between the corrosive environment and metal surface, restricting the corrosive agents (oxygen, water, and ions) to diffuse to the metal surface. [5] With this intent, polymer composites are pointed out as promising materials to be used in the protection of automotive components and have shown to improve the mechanical, tribological and anticorrosive properties of metal substrates. [6] Together to polymeric resin (e.g. epoxy, polyurethane, polyester), different types of pigments, fillers, inhibitors, and other additives are employed to fabricate high-performance protective systems. High barrier level is reached by reinforcing the coatings with inorganic fillers as transition metal sulfides, ceramic nanoparticles, soft metals, mineral silicon salts, and most recently, graphene. [7]

In great evidence since it was prepared for the first time in 2004, graphene is classified according to the number of layers, dimensions, structural rearrangement, and number of oxygens present in the structure, e. g. single-layer graphene (SLG), multi-layer graphene (MLG), graphene oxide (GO) and reduced graphene oxide (rGO). [8] Outstanding mechanical resistance, high surface area and electric conductivity are among the most recognized graphene properties. [9] In view of that, graphene structures can be used in a range of applications such as textile industry [10], energy storage [11], optoelectronics [12], as well as protection against corrosion [13]. Graphene physicochemical properties and its nanostructured morphology contribute to the formation of isolation layers in graphene composite coatings. This hydrophobic and electrically charged layer structures can postpone or even prevent water/corrosive ions from penetrating into metal substrate, delaying the rate of corrosion. In this paper, we evaluated the potential use of protective coatings based on graphene/polymer composites in the automotive industry, considering some of the latest advances in the corelated scientific research, industrial solutions, and commercial products.

Graphene Reinforcement to Automotive Composite Coatings

Besides providing esthetical finishing, coatings are crucial for reducing mechanical wear and improving corrosion resistance of car components. Typically, automotive painting comprises four layers: e-coat, primer, basecoat and clearcoat, as illustrated in the Figure 1. In general, after a pre-treatment, which includes cleansing and phosphatization processes, the e-coating is applied by electrodeposition to provide corrosion resistance to automotive parts. Next, the primer is intended to aggregate properties, such as resistance face to UV radiation and stone chipping, as well as yielding a more regular painting interface previously to the application of the topcoat. The topcoat comprises the basecoat, which display the main role in the appearance of the coating, and the clearcoat, a sealing transparent layer employed to preserve the coating from weathering. [14]

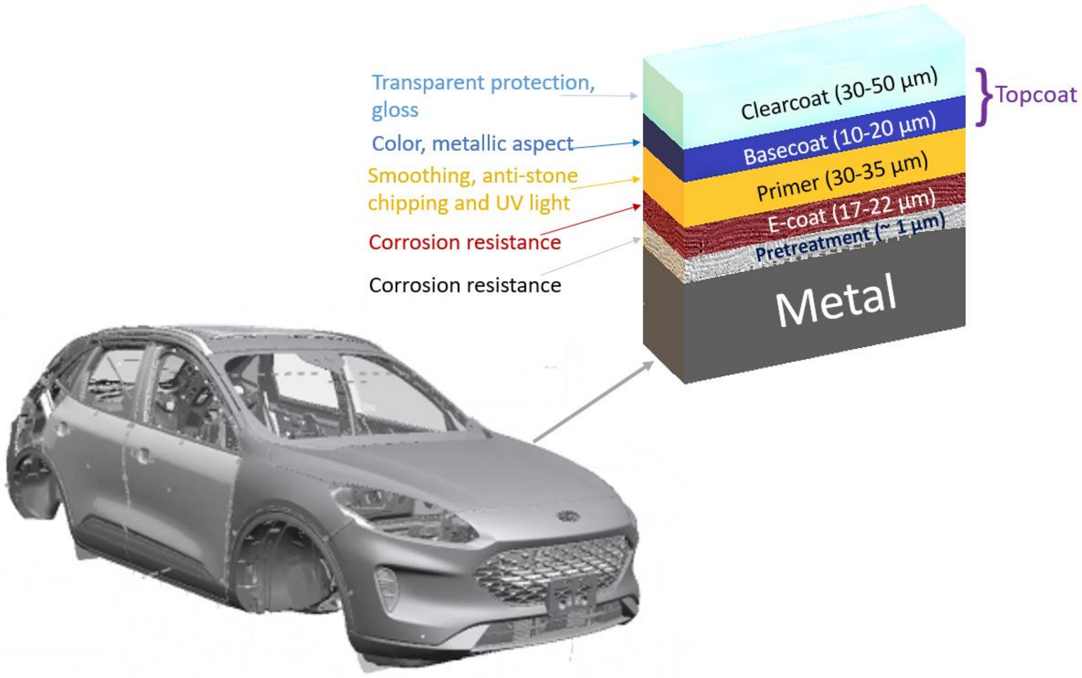

Figure 1: Composition of automotive painting.

Among automotive coatings, composites have been pointed as advanced materials able to match the modern vehicles specifications related to physical, chemical, and electrical properties. [15] In EVs for example, the use of these materials could impart significative improvements in corrosion resistance and tribological properties [16], besides other indirect advantages as thermal and mechanical stability, and weight reduction of car components. In this context, we have conducted a survey in Patentscout platform (Jun/2022), including specific keywords found in the abstract (automotive, composite coatings), which returned 860 active patents. Using the *text cluster* tool, from the same website, a subclassification of the patents were obtained, resulting in

241 patent groups (Figure 2), aggregated mainly according to the material composition and coating functionality.

AUTOMATIVE OEM 9.4 %	Automotive OEM finishing	Cross-linking agents
	Prediction curve	Gloss
	Functional groups	Acrylate monomers
CLEAR COATING 9.2 %	Multilayer coating	Glass transition temperature
	Hydroxyl groups	Organic solvent
	Polyurethane coating	DIN 53240
POWDER COATINGS 7.7 %	Epoxy resins	Effect pigment
	Polyester resin	Metal powder
	Automotive parts	Corrosion resistance
COATING MATERIAL 33.2 %	Automotive interior	Plastic substrates
	Powder coating compositions	Epoxy resins
	Clearcoat materials	High strength
COATING FILM 19.8 %	Acrylic resin	Multi-layer coating
	Film layer	Chemical resistance
	Automotive interior	Effect pigment
METALLIC COATING 20.7 %	Protective coating	Target coating
	Film forming	Corrosion resistance
	Fine-grained metallic coatings	Non-metallic coating

Figure 2. Patents subclassifications (241) related to the Patentscout search, using the keywords: automotive, composite coatings (jun/2022). [17]

Despite this significant number of results, when the word graphene was added to the keywords used previously, only 6 active patents were found in Patentscout. Nonetheless, graphene containing coatings display a promising alternative for enhancing the performance of the current composite coatings. In particular, the use of graphene is appropriate to develop features related to two recurring aspects mentioned in the clusters: corrosion resistance and clearcoat performance.

Its exceptionally high surface area and electronic conductivity (resulting of the sp^2 hybrid structure) turns graphene a suitable material to act as both physical and electronic barrier to the passing of corrosive agents, such as water molecules, oxygen, and negative ions. In view of that, many studies have discussed the use of graphene combined with polymers in composite coatings [18]. However, since graphene has a non-polar molecular structure, its interaction with organic resins, usually rich in polar sites, is a critical factor for the obtention of efficient composite coatings. One of the most explored routes to improve this interaction is through the chemical transformation of graphene in graphene oxide (GO) or reduced graphene oxide (rGO). This oxidation yields polar sites in the graphene structure, which can interact with the organic groups of the resins. Furthermore, the presence of oxygen-containing functional groups also enables the utilization of GO and rGO in waterborne coatings, since hydrophilic groups are introduced by oxidation. [19] Besides this modification, it is common the use of coupling agents to ensure a better dispersion of graphene or GO along polymeric coatings. Basically, these agents have both functional groups, which have good interaction with the filler, and others which can form chemical bonds with the polymer, acting as anchoring sites for the additives.

An example of these composites is shown in the work of Zhu et al. [20], in which an epoxy resin obtained from cardanol is used to improve GO dispersion in a commercial epoxy. X-ray photoelectron spectroscopy (XPS) analysis confirmed cardanol contribution to GO dispersion and according to electrochemical impedance spectroscopy (EIS), this composite coating has shown excellent anti-corrosion performance with high corrosion resistance (up to 10^8 Ω.cm^2), after 45 days of testing. Similarly, Mo et. al. investigated the use of graphene and GO to improve mechanical properties of a polyurethane (PU) coating. For a better dispersion, the fillers were reacted with 3-amino-propyltriethoxysilane (APTES) yielding functionalized graphene (FG) and functionalized GO (FGO). Raman spectroscopy, X-ray diffraction (XRD), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) proved the efficiency of the chemical modification in facilitating the dispersion and compatibility of fillers. By adding FG and FGO in the range of 0.25% to 0.5% tribological and anti-corrosive properties of PU were enhanced. However, these properties were worsened with a larger amount of the filler, which was attributed to the growing number of cracks across the composite. Finally, the results pointed that FGO/PU exhibited better tribological property, but worse anti-corrosion performance compared to FG/PU. The reason for that would be the presence of the oxygenated groups of GO, which represent more reaction sites for the functionalization (and thus a better dispersion), but also could damage the graphene lattice structure, weakening its barrier capacity. Other authors have shown that reinforcing polyester coatings with GO and rGO, in the range of 0.3 wt % [21] to 1% [22], has also improved tribological and anti-wear properties. Still, most researchers have developed graphene composites with epoxies. These resins stand out among polymer coatings for their preeminent resistance against corrosion [23]. Table 1 presents examples of scientific works published using epoxy-based paints deposited on steel, in which graphene and its derivatives were used additives in the paint composition. All papers have shown increased corrosion resistance with the incorporation of graphene structures to the epoxies.

Table I: Summary of epoxy-based paints with graphene and derivatives.

Graphene Type	Filler Content wt%	Resin Type	Coupling/Additive Agent	References
GO	(0.5%)	siloxane-epoxy resin (SILIKOPON EF)	Silane (TEOS)	24
GO	(1.0%)	Waterborne epoxy (resin MU-601)	1-aminoethyl-3-methylimidazolium bromide (Ionic Liquid)	25
GO	(0.1%)	Araldite (GZ7 7071)	Silane (TEOS and APTES)	26
GO	(0.2%)	Bisphenol A / Epichlorohydrin (EPON 828)	Oxalyl chloride (COCl)	27
GO	*	Bisphenol A / Epichlorohydrin (EPON 828)	Polyaniline (PAni)	28
GO	(0.1-5%)	Diglycidyl ether bisphenol A, (Epon 828)	p-phenylenediamine (PPDA)	29
GO	(1.0-4.0%)	Bisphenol A (E44)	Polyaniline (PAni)	30

GO	(0.5%)	Bisphenol A / Epichlorohydrin (EPON 828)	4-nitroaniline	31
GO	(1.0-4.0%)	Bisphenol A / Epichlorohydrin (EPON 828)	3-amino-1, 2, 4-triazole-5-thiol (ATT)	32
GO	(0.1-0.25%)	Araldite	4-fluorophenol	33
GO	(3.0%)	Araldite (GZ7 707)	APTS	34
GO	(0.2%)	Bisphenol A / Epichlorohydrin (EPON 828)	Polyaniline (PAni)	35
GO	(0.1-1.0%)	Bisphenol A (E44)	2-(3,4-epoxycyclohexyl)ethyl triethoxysilane (ETEO)	36
GO	(0.1%)	Bisphenol A (E51)	Silane (APTES)	37
GO	*	Waterborne epoxy Zn-Al coatings	Silane (APTES)	38
GO	*	Bisphenol A / Epichlorohydrin (EPON 828)	Hyperbranched polymer (HBP)	39
GO	(0.1-0.7%)	Waterborne epoxy latex	Polyvinylpyrrolidone (PVP)	40
GO	*	Waterborne epoxy	polypirrole (PPy)	41
GO	*	Araldite	2- Aminothiazole (AT) and 2-Amino-4-(1-Naphthyl)Thiazole (ANT)	42
GO	(0.25%)	Waterborne epoxy (H228A)	tea polyphenol	43
GO	(1.0%)	Bisphenol A diglycidyl ether (Epotec YD-120)	polyamide	44
GO	*	Bisphenol A diglycidyl ether	Polyaniline (Pani) and chitosan	45
Graphene	(0.5%)	F0704 - waterborne (WEP)	Perylene bisimide (PBI)	46

Graphene	(1.0%)	Bisphenol A (E44)	-	47
Graphene and GO	(0.1-0.5%)	Bisphenol A (E51)	-	48
Graphene and GO	(0.38-0.89%)	Bisphenol A (E51)	Silane (KH-550)	49

As shown, most of the composite presented have been formulated using GO, less prone to agglomerate in the coating. Although the addition of pure graphene to resins is still limited by issues related to dispersion, some patents have purposed strategies to prevent the formation of graphene clumps in the matrices through industrial methods as high-shear convection dispersion [50] and gas grinding [51], enabling the processing of these composites. Thus, the need to oxidize graphene could be suppressed for large scale processes, however pure graphene production cost is still a relevant concern. Fortunately, cost-effective graphene production techniques have evolved, as in the case of top-down methods based on graphite bulk exfoliation. [52] Moreover, low filler content is usually present in graphene composite (see Table 1), so the impact of this additive on the final cost of the coatings would not be very compromising.

Up to now, only few examples of graphene containing coatings are already commercially available. [53] These products are basically graphene reinforced ceramic composites acting as clearcoats. Such formulations are developed to provide superior resistance to abrasion, etching, stone chipping, scratching, besides reducing water-spots on the car basecoats thanks to graphene mechanical resistance and hydrophobic character. [54, 55] Another advantage claimed is the reduction in the heat absorption, which can be connected to the graphene's ability to improve thermal dissipation on metals [23]. By providing protection against a variety of external degrading agents, these coatings aim to enhance the durability of topcoats by at least 10 years. [53] Figure 3 summarizes some of the main topics involving the graphene reinforced coatings technology.

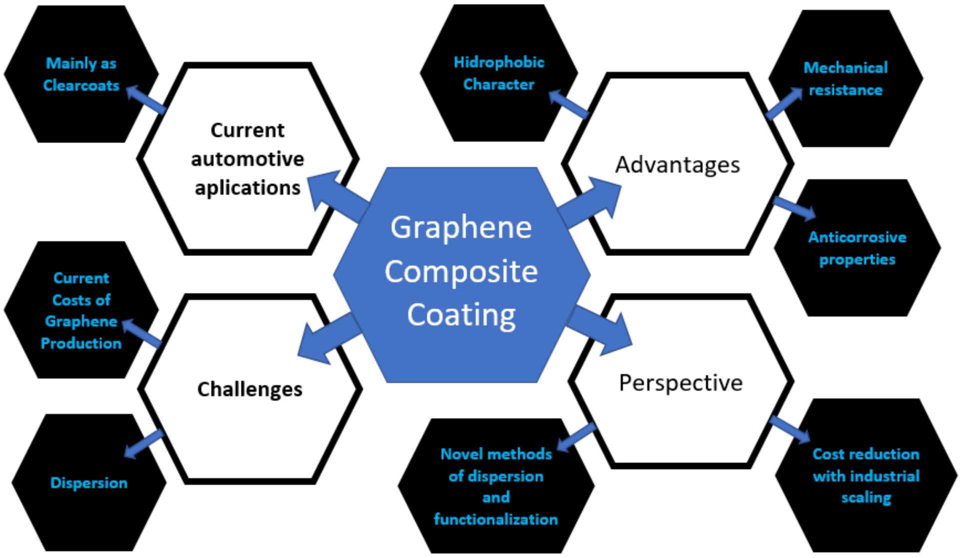

Figure 3: General panorama of graphene composite coatings.

Summary and Next Steps

The present paper provided a general view about the current state of art and uses of graphene in the protection of metallic substrates. The reinforcement of typical polymer resins with graphene, GO or rGO have presented some expressive results in coatings, with better anticorrosive properties, besides improved resistance face to mechanical and environmental damage. The dispersion and interactions of graphene structures along the resins are still the main challenges in processing of these composites. Nonetheless, with the growing interest in nano-additives, innovative (or combined) methods of dispersion and the use of coupling agents have been purposed to make more feasible the production of these coatings in industrial scale. Hitherto, commercial automotive graphene coatings are quite restricted to ceramic topcoats. However, its versatility and noticeable results reveal the capability of graphene composite coatings to meet the demanding protection standards of the new car's generation.

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