Char Strength of Wool Fibre Reinforced Epoxy-Based Intumescent Coatings (FRIC)

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Abstract. Fire protective intumescent coating cannot insulate a base material effectively if its char lacks mechanical strength. This research therefore, studied the effects of fibre reinforcement to epoxy-based intumescent coating's char strength. The fibres used include glass wool fibre, Rockwool fibre and ceramic wool fibre of 10mm length. The three formulations' mechanical performances were compared to both, a famous commercial intumescent coating and a control formulation without fibre. These coatings were fire tested up to 800°C in an electric furnace for an hour. Their chars' mechanical properties were evaluated for char resistance test using predetermined weight loads. In the test, masses from 100g to 3600g were loaded continuously on top of the chars where the fibre reinforced intumescent coating (FRIC) has shown better strength and resistance to deformation. As a result, they produced lower percentage of height reduction i.e. 34% - 83% different when compared to unreinforced coating. Control char also ruptured at as low as 4N load. It was deduced that fire insulative wool fibres are effective reinforcement for improved char strength of the FRIC.

Introduction

The worldwide fire protection industry is estimated over US$50 billion in 2008 with a growth in industrial fire protection of 11 percent [1]. Intumescent coating is a trusted passive fire protection where its application for example in the offshore industry grows rapidly [2] after the Piper Alpha disaster [3]. Conventional intumescent coatings have soft char and hence their installation require wire or fibre mesh.

Fibre reinforcement is a breakthrough finding that enhances mechanical properties of composite materials [4]. A reinforced intumescent coating would have more compact cell structure and of higher strength. This eliminated the use of external mesh that in return reduce the works in applying protective coatings and therefore more economical. Wool fibres are fire retardant, insulative, cheaper and are in abundance than other specialized fibres. Previously, in this research it was reported that glass wool fibre, Rockwool fibre, ceramic wool fibre, carbon fibre, glass fibre, chopped fibre strand and hybrid fibre reinforced intumescent coatings (FRIC) produced char of better structure and thermal properties than the control (without fibre) in both 400°C and 800°C fire tests [4-5]. However, the mechanical properties including the strength of intumescent coating chars, which are vital to combat the forces of fire, have not been thoroughly studied. There is a technique devised by a group of researchers using a modified furnace rheometer to test the mechanical resistance of the intumescent coatings' char in a closed space until only 1600g compression force [6-7] or about 16N.

The unique contribution of this study is the comprehensive mechanical properties assessment and their test methods of FRIC chars. The aims here are to study and compare mechanical properties of FRIC to control formulation and a commercial coating loaded to 3600g mass or approximately 36N force. Report of such study is yet available. The discovery will support the theory of better mechanical properties of char, expected by fibre reinforcement to intumescent coatings and justify the char strengthening mechanism proposed [8].

Experimental Details

Materials and Formulation Preparation. A control intumescent coating formulation, mainly consists of ammonium polyphosphate (APP), pentaerythritol (PER) and melamine (MEL) and three FRIC formulations (control + fibre) have been coated onto different mild steel plates. In each studied formulation, there were 15 ingredients including fibre. Intumescent ingredients and their mixing technique were previously elaborated [4]. A commercial intumescent coating (Chartex 7) was also prepared for comparison purpose. Samples details are shown in Table 1. Approximately, 20g of coating was evenly applied with metal spatula onto a 50mm × 50mm × 1.5mm mild steel plate supplied by TSA Industries (Ipoh) S.B. readily coated with primer coating (Dulux Epoxy-Zinc Phosphate). The coatings were left to dry at ambient temperature. Dry coating thickness was measured using Mitutoyo digital thickness gauge were 4.5mm and 7.0mm for the new coatings and Chartex 7, respectively. It is known that it is difficult to get a thinner coat for the commercial coating.

Fire Test. The coating samples were exposed to high temperature fire test and their chars were evaluated for char strength by char resistance test. In the fire test, progressive heating with the rate of 26°C/min from room temperature to 800°C was the best achievable with the model CWF 1300 Carbolite furnace to follow the standard temperature/time curve in BS 476-20. The temperature was held for an hour before cooling down to room temperature.

Char Resistance. This test determined quantitatively the char strength of intumescent coatings by studying char ability to resist deformation at specific loads. This was done by gradually and continuously adding load on top of a char at 100g increment until 3600g load. Seven disc-type loads of known mass; 100g (1 piece), 200g (2), 500g (1), 600g (1) and 1000g (2) were used. At each load, after a minute the height of the char was recorded. Shown in Fig. 1 is the experimental setup.

Table 1 Intumescent coating test pieces descriptions

Formula	Test Pieces	Fibre	Average Diameter (μm)	Length (mm)
1	GWFRIC	Glasswool fibre	17.3	10
2	RWFRIC	Rockwool fibre	4.7	10
3	CWFRIC	Ceramic wool fibre	2.8	10
4	C7	Mineral fibre & Al_2O_3/SiO_2 fibre	6.5 & 1.5	« 1
5	Control	Nil	NA	NA

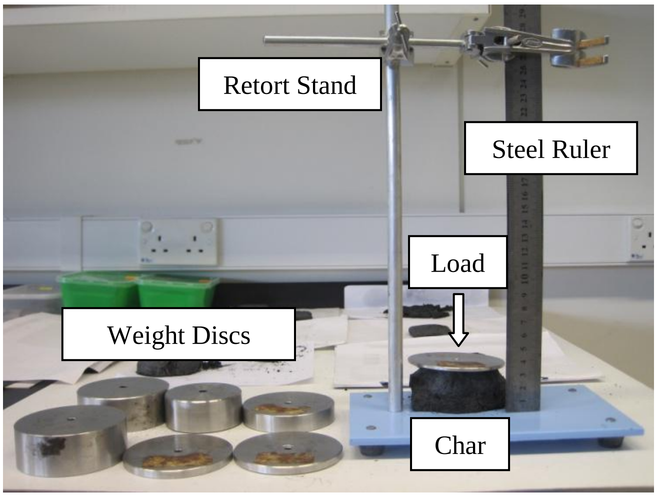

Fig. 1 Experimental setup for char mechanical resistance test

Results and Discussion

Fire Test. Furnace fire simulates a fire incident in a confined space, where intumescent coating samples were allowed to intumesce freely. A combination of large, thick and homogenous char [9], in addition to closed-packed structure gives better fire protection due to its effectiveness in restricting heat transfer to substrate [10-11]. These physical findings of the chars produced after 800°C fire test will be correlated to the char strength. This temperature is also not far from the standard test curves (maximum 1000 – 1200°C) i.e. BS 476-20 [12] and UL 1709 [13]. The purposes of adding fibre are numerous [14], among them are to reinforce or strengthen the char formed from intumescent coating, and to improve the insulation of the coated substrate and durability of the char. Mineral fibre for instance mineral wool fibre that is fibrous in nature, provides good mechanical structure to the material it is reinforcing [15]; in fire it develops phosphorosilicate glass within the char [14]. High temperature fillers [4] were added into the formulations to form a compact microstructure in the charred layer [11] and stabilize the char [16]. Without additives or reinforcement, old intumescent coatings consisting APP/PER/MEL are known to produce a fluffier barrier of fire retardant, which is easily penetrated by fire [11]. Adding epoxy binder is known to impart better fire protection [17]

Table 2 gives the physical characteristics of the chars produced for this study. FRIC chars showed higher growth than the commercial one, C7. From literature, C7 contains commercial fibres; amorphous mineral fibre and high surface area alumina silica fibre, and at least fifteen other proprietary ingredients [14, 18]. However, control char from an unreinforced formulation again showed the most expansion [4]. It is followed by GWFRIC but inspection at its cross-section char piece revealed many voids suggesting substantial amount of gas released from the char in fire, which promotes char growth. Moreover, glass wool fibre has lower density that gives small restriction to char expansion than the other two fibres studied. On the other hand, CWFRIC produced the most char yield and lowest char weight loss. Weight of a char reduced as compared to its coating due to materials lost in fire. The greater is the weight loss the smaller is the available amount of materials to protect the substrate [4]. Flame retarded wool fibres have been successfully applied with intumescent materials to develop flame retardant textiles [19-20]. In the present research, the new wool fibre-reinforced formulations had less weight loss and therefore produced more char or residue that refrain flame than the benchmark coating.

Char Resistance. This is a simple test created to quantitatively measure char performance in term of its strength to hold load. The strength was quantified relatively by the measurement of the char height after the load was introduced. A stiffer char will resist deformation to its structure harder and this was translated by smaller change in the height of the char. The sooner a drop in height was detected, possibly the lower is its strength. An expansion of the control char was not restricted since its char is full of holes, which acted as the air passage while the blowing agent is reacting in a fire. The char structure was not suppressed and connected by fibres. As a result, the weakest char was produced. Shown in Fig. 2(a) is the exponential-shape plot of the control char as it is loaded with weights up to 3600g. It ruptured at load of 400g, confirmed by the sudden drop of the char height. At about 1700g, the change in the height was minimized and a plateau was developed as the load increased.

Oppositely, FRIC and the market coating did not follow the exponential-like graph due to little change in the char height experienced as shown in Fig. 2(b). This observation can be attributed to the fact that these coatings are highly crisp [4] or brittle, possessed only tiny holes and therefore are of higher strength. Nevertheless, glass wool fibre formulation endured more than half height reduction of that by the control. This showed that the char being the second highest was compromised by the lower char yield it produced compared to its counterparts; Rockwool fibre and ceramic wool fibre.

Table 2 Physical performances of the chars produced after 800°C fire test

Sample	Char Physical Properties				
	Growth	Char Yield (%)	Wt Loss (%)	Colour	Adhere to Substrate
GWFRIC	489% (5.89X)	36.9	63.1	Black	Yes
RWFRIC	300% (4.00X)	43.8	56.2	Black	Yes
CWFRIC	233% (3.33X)	46.1	53.9	Black	Yes
C7	86% (1.86X)	34.5	65.5	Black	Yes
Control	669% (769X)	33.5	66.5	Black	Yes

Fig. 2(b) also shows CWFRIC had only a single change in the char height, which occurred at a load of 2500g. While the first reduction in height from RWFRIC and C7 happened at 1600g and 1200g, respectively. The first drop in thickness due to the compression force may be attributed to collapse or rupture of the rough peak of the char's top layer. Successive reduction in char thickness happened because of further break of the top surface. Displaying slightly distorted straight line plot, these three formulations did not rupture at the maximum test load of 3600g. This is evident in Fig. 3(a-c) as CWFRIC sustained the loads, undamaged. Whereas, GWFRIC exhibited softer behavior with half of its original height was compressed at load of 3300g. The highest height reduction was recorded at 86% by control char; see Fig. 3(d). In summary, FRIC gave 34%-83% lower drop than the control.

The increase in mechanical strength of the fibre reinforced chars was anticipated. Though, high temperature may have decreased fibre performance, FRIC produced higher strength chars than the unreinforced coating. Ceramic wool and Rockwool fibres of greater length also performed better than short fibres in C7. These showed that fire resistant fibres such as carbon fibre, Rockwool fibre and ceramic wool fibre are good candidates for a highly insulative and strong char.

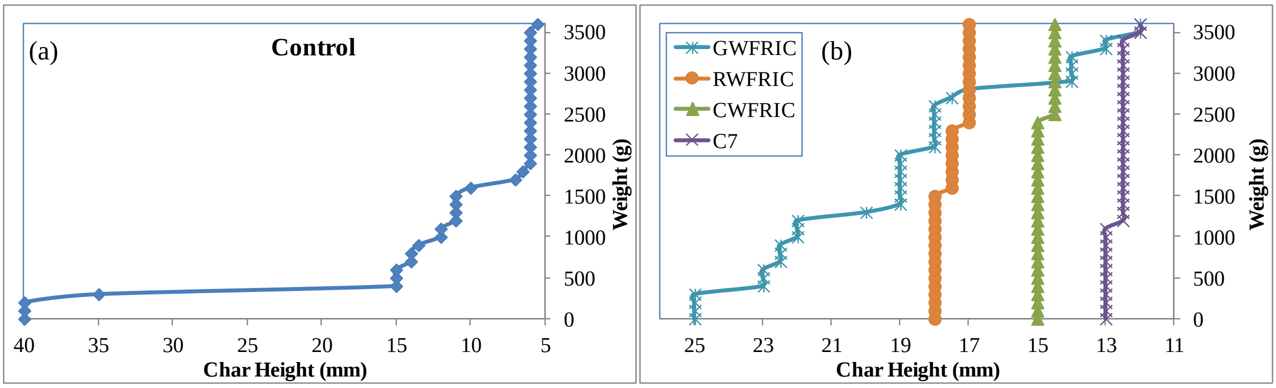

Fig. 2 Mechanical char resistance of (a) control, and (b) different formulations after 800°C fire

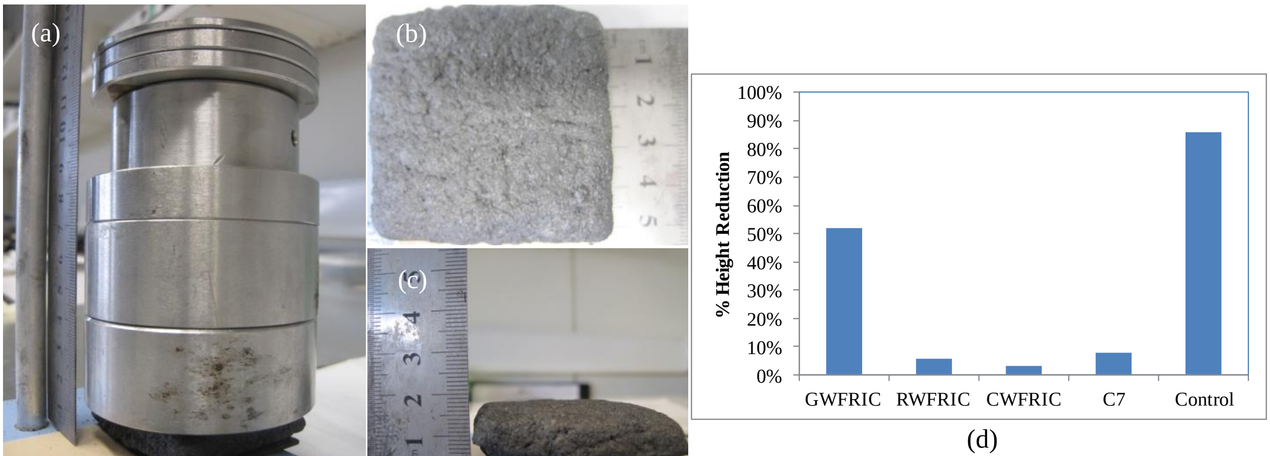

Fig. 3 The digital camera images of CFRIC char (a) under 3600g loads, (b) top view after test, (c) side view after test, and (d) percentage in height reduction after char strength test

Conclusion

A fire protective intumescent coating will not be able to insulate a base material effectively if its char lacks mechanical strength. In this research the char strength of FRIC were studied and compared to the chars from a control and a commercial formulations. For this reason, a new method of experiment was devised. The findings from this research have supported the theory of better mechanical properties imparted to intumescent coating formulation by fibre reinforcement. FRIC coatings as being reinforced with fibre have demonstrated better mechanical properties with CWFRIC became the top performer. Fibres, being highly insulative, high temperature resistant and strong have contributed a great deal in producing intumescent coatings with their chars of better mechanical properties. Also shown was the suitability of the test methods to quickly and accurately measure mechanical properties of a char.

Acknowledgements

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