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Mechanisms of Char Strengthening in the Fibre Reinforced Intumescent Coatings

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Abstract: Intumescent coatings are used to protect steel structures from fire attack. However, soft chars from traditional formulations make the protection less effective. The aim of this research was to study three formulations of Fibre Reinforced Intumescent Coatings (FRIC) using various types of wool fibres. Fibre reinforcement was introduced to strengthen the coatings and their chars for better fire protection to the substrate. The FRIC coatings were fired to 800°C for 1 h duration in a furnace to produce fibre reinforced chars. Examination using Field Emission Scanning Electron Microscopy (FESEM) was conducted on the coatings and their chars for morphology study. These scans explained the roles played by the fibres, where a number of mechanisms in strengthening the chars were observed. Energy Dispersive X-ray (EDX) Spectroscopy analysis between the coatings and their chars showed that FRIC was able to sustain high temperature materials and therefore was considered to have rise the char strength and fire protection. These findings concluded that fibre reinforcement strengthened FRIC chars in four mechanisms.

Key words: Intumescent coating, fibre reinforcement char, fire protection, wool fibers

INTRODUCTION

To control fire hazards effectively one can relies on excellent reactive passive fire protection such as intumescent coating. This coating if applied to the structure of a building will retard fire and slow down the spread of flame. However, the existing intumescent coatings are expensive and lack char strength (Amir *et al*., 2011a, b). Mechanical properties of a char is therefore a key parameter, so that the char must not be easily destroyed by external perturbations for a coating to deliver full protection (Jimenez *et al*., 2006).

The aims of this study were to characterize three Fibre Reinforced Intumescent Coatings (FRIC) using 10 mm long glass wool fibre, rockwool fibre and ceramic wool fibre. Analytical approaches were adopted to correlate the findings in determining the strengthening mechanisms of the fibres in the char. Wool fibre such as Rockwool fibre, which is a mineral-based and fibrous, provides good mechanical structure to the material it is reinforcing. This fibre together with silicon- containing fibres, in fire develop phosphosilicate glass within the char enhance the insulating properties and durability (Hanafin and Bertrand, 2000). Furthermore, these fibres are much cheaper than other specialized fibres and are in abundance. Epoxy-amine composite was shown benefited better thermal properties by adding thermally stable fumed silica (Sipaut *et al*., 2007).

This is the third publication by the author on scarcely reported FRIC and the second part of research on FRIC using wool fibre. Wool fibre-FRIC was shown to perform better than a commercial coating; greater char expansion, lower weight loss and higher char yield, better char structure and morphology with almost identical compounds were developed suggesting good fire performance (Amir *et al*., 2011a). The fibres used either undamaged in the fire or slightly deformed. The new intumescent coatings formulations being reinforced with various types of long wool fibres, which were normally used as insulation have been shown to improve coating's char properties including their strength by producing more char and having denser structure (Amir *et al*., 2011a). Earlier studies on four types of FRIC had also demonstrated better intumescent char characteristics when compared to an intumescent formulation without fibre (Amir *et al*., 2011b). Also, shown were the long carbon fibre and glass fibre formulations developed greater char's height, promoted more char formation with denser cell structure for improved char strength.

Unique contribution of this study is the proposed strengthening mechanisms to better understand fibre roles as reinforcement for future manipulation. Direct

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reinforcement of fibre eliminated usage of external mesh to reduce the works in applying protective coatings hence, more economical. Fibre strengthening mechanisms in the char explained the strength enhancement anticipated for the FRIC.

APPROACH AND METHODS

Materials and formulation preparation: Three new FRIC formulations have been prepared; glass wool fibre reinforced (GWFRIC), Rockwool fibre reinforced (RWFRIC) and ceramic wool fibre reinforced (CWFRIC), and coated onto mild steel plates. Their descriptions are shown in Table 1. Intumescent ingredients consist of three main components (Wang and Chow, 2005), at 3:1:1 ratio of ammonium polyphosphate/pentaerythritol/ melamine. In each formulation, there were 15 solid or liquid ingredients including fibre. The insulative wool fibres were supplied by FOSTER (M) S.B.

Using metal spatula, approximate of 20 g coating was evenly applied onto a mild steel plate of dimension, 50×50×1.5 mm 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 for several days. Dry coating thickness was measured using Mitutoyo digital thickness gauge giving 4.5 mm. Figure 1a shows the CWFRIC sample.

Fire test: Constant heating rate of around 26°C min^{-1} from room temperature to 800°C within 30 min in Carbolite electric furnace closely followed the standard temperature/time curve in BS 476-20. The temperature was then maintained for another 30 min before the samples were taken out and left to cool down. A typical char sample for CWFRIC is shown in Fig. 1b. Physical properties of the chars were measured; height, weight, crispness, shrinkage and cell structures after manually cutting through Fig. 1c were examined.

Field emission scanning electron microscopy: Appropriate samples were prepared by cutting a small portion across the coatings and their chars (through thickness) for morphology study, as in Fig. 1d. Scanning electron was performed using field emission SEM (FESEM) ZEISS SUPRA 55VP, operated by Electron High Tension (EHT) in the range of 15-20 kV, at 8.0 mm working distance and using VPSE signals. The magnification was maintained at 500x.

Energy dispersive X-ray spectroscopy (EDX) analysis: SEM-EDX analysis was also run using the FESEM machine that provides rapid qualitative and quantitative

Table 1: Test pieces and their fibre information

Sample	Fibre	Length (mm)	Diameter (μm)
GWFRIC	Glass wool fibre	10	17.3
RWFRIC	Rockwool fibre	10	4.7
CWFRIC	Ceramic wool fibre	10	2.8

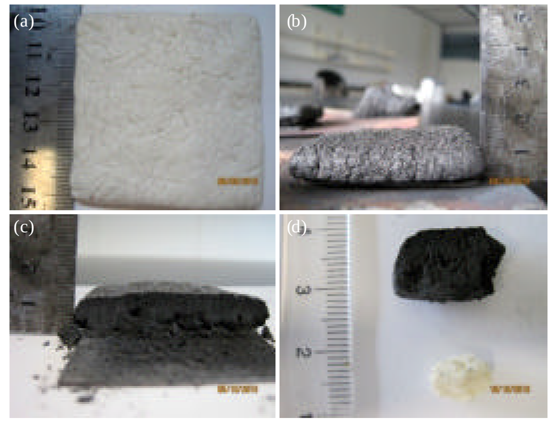

Fig. 1(a-d): CWFRIC sample preparation for fire test and SEM test and SEM-EDX analysis, (a) Coated sample, (b) Char (c) Char cross-section and (d) Top-view of through thickness coating

analysis of elemental composition. In the area scan, spectrum 0-10 keV processing, no peaks omitted and processing option was set to "All elements analyzed (Normalised)" under four numbers of iterations.

RESULTS AND DISCUSSION

Fire test result on the physical properties of the chars were discussed previously (Amir *et al*., 2011a). They performed better than a commercial coating in term of lower weight loss (more char) (Gu *et al*., 2007), good expansion and strong, denser char structure. Effective protection to the steel substrate is also determined when the chars are not easily damaged by outside factors (Jimenez *et al*., 2006), which is proven by the FRIC chars.

FESEM examination: It was however, not easy to obtain a clear view of full fibres in the micro-size scan of a coating sample. This is because, the fibres, which were considerably long were 'hidden' and coated with intumescent ingredients and binder materials after mixing and cured. Trained and experienced machine operators need also to skillfully adjust the scanning to eliminate as much possible the effect of charging image of elements especially in chars for every micrograph prepared so that its quality was not compromised.

Figure 2 shows examples of these fibres, which were intact and fully coated or immersed in the coatings. After mixing the fibres mostly settled horizontally due to gravity force throughout the coatings. It was obvious that when intumescent coating cured, these wool fibres provide mechanical structure to the coatings and act as 'linkages' to hold coating's ingredients together for a stronger bond.

Further analysis of the char micrographs with few examples as exhibited in Fig. 2 revealed that the wool fibres reinforced the char microstructure by becoming barriers (Hanafin and Bertrand, 2000) for holes formation, which were normally created when the blowing agent react against fire in expanding the voluminous carbonaceous char and when the cooling gases were released. Big voids are usually associated to low-strength

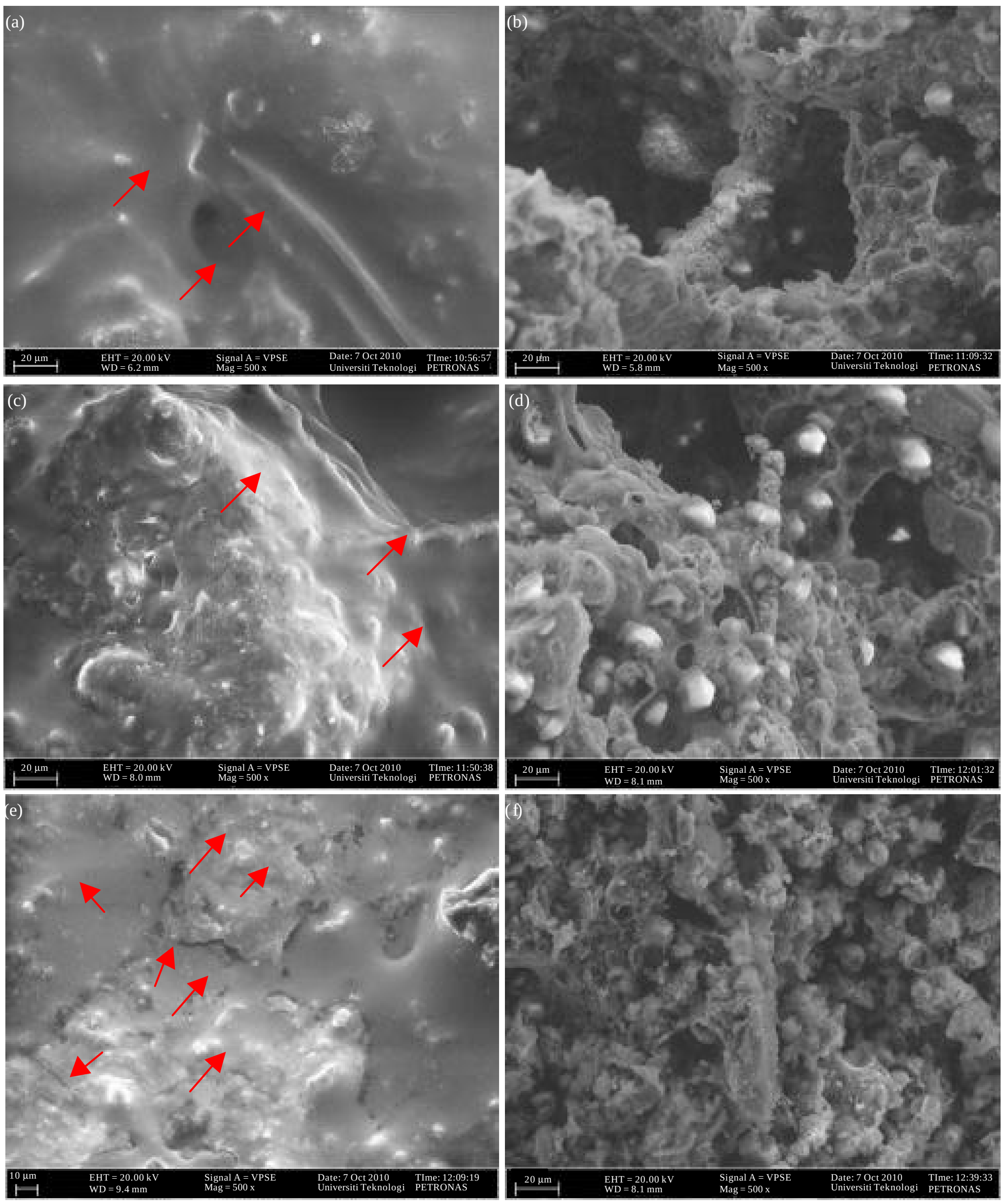

Fig. 2(a-f): SEM images across the thickness cross-section at 500x magnification of (a) GWFRIC coating, (b) GWFRIC char, (c) RWFRIC coating, (d) RWFRIC char, (e) CWFRIC coating and (f) CWFRIC char with arrows showing the location of fibres

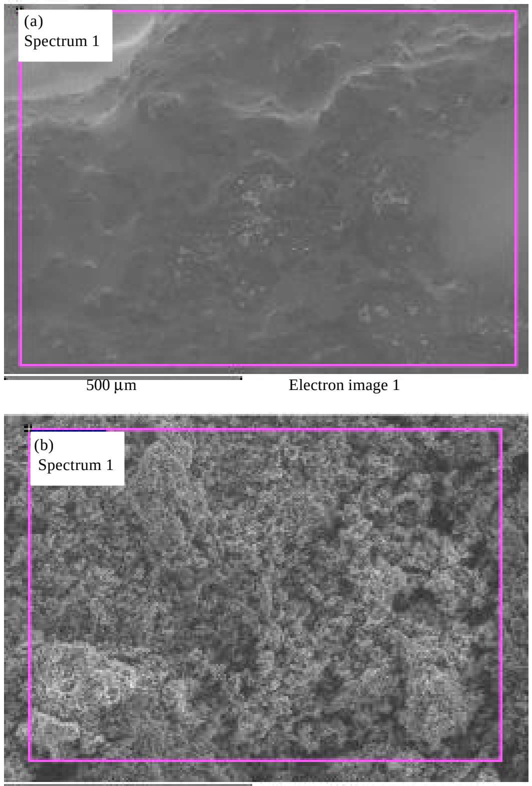

Fig. 3(a-b): SEM-EDX area scan of CWFRIC (a) Coating and (b) Char

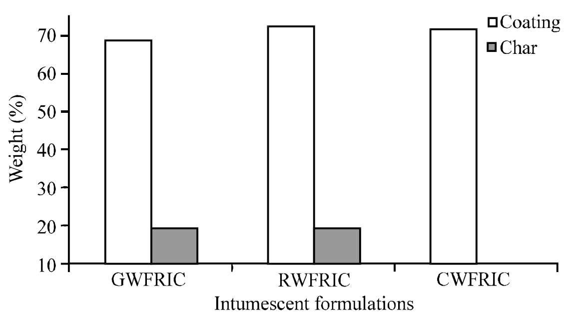

Fig. 4: Carbon weight for the coatings and chars

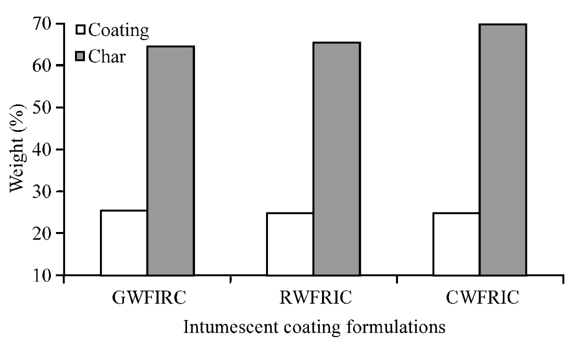

Fig. 5: Oxygen weight for the coatings and chars

char and reduced fire protection. Fibre barrier assists in forming smaller micro-voids that renders stronger and denser structure, and are also effective in trapping air for increase insulation properties to the char.

The second mechanism is when fibres provide good mechanical structure to the char, Fig. 2. Fibres preferentially form link or bridge with other intumescent materials, a situation called as char bonded (Kandola *et al*., 2001), result in closed packed structure char. The fibres were fully coated in the coating mixture and were effective to hold coating's ingredients to prevent fire penetration.

Thirdly, fibres become char promoter (Kandola *et al*., 2005) and build up char volume. For instance development of fire retardant glass or crystals on the fibre surface at high temperature i.e., glass fibre (Amir *et al*., 2011b), glass wool fibre, and Rockwool fibre (Fig. 2b-d). In the fourth strengthening mechanism, fibres that are non-combustible retarded fire. These fibres do not degrade or deform at high temperature, therefore maintaining high residue, less weight loss i.e., carbon fibre, ceramic fibre (Amir *et al*., 2011a) and ceramic wool fibre (Fig. 2f).

Comparatively, ceramic wool fibres gave better physical obstruction. The fibre promoted crowded char structure as evident in by filling up bigger pores and reduced the size of microscopic voids. In return, the number of effective cavities to be filled by air were increased that enhanced char insulation.

EDX analysis: Shown in Fig. 3 are the SEM-EDX scans of CWFRIC's coating and char. The samples when compared provided partial similar trends. Figure 4 and 5 show increment by weight of Oxygen (O) and reduction of Carbon (C) as coatings transformed into char in fire. Among the coatings, the highest C content and the lowest O content belonged to RWFRIC. It was followed by CWFRIC and GWFRIC for C content, whilst for O content; GWFRIC was the highest, then CWFRIC. CWFRIC char showed the highest weight of O element as may also partially contributed by ceramic fibre, which is rich with oxygen, followed by RWFRIC and GWFRIC. Oppositely, for C content among the chars; the rank was RWFRIC>GWFRIC>CWFRIC.

There were increment shown in Phosphorous (P) and silicon (Si) and reduction of calcium (Ca) weight from the coating to the char form as given in Fig. 6 and 7, respectively. The exception was for GWFRIC, where Si high content was almost maintained. Even though P element, which is an important fire retardant intumescent material, was detected lower in the FRIC, the produced P by weight in their chars was relatively much higher suggesting huge potential of fire protection

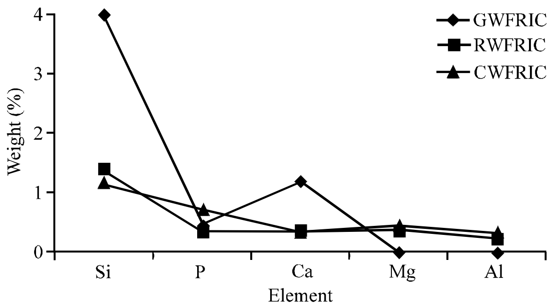

Fig. 6: Element weight for the coatings

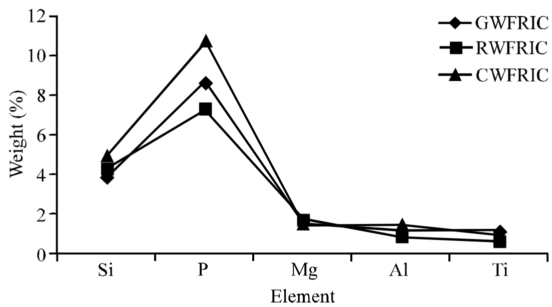

Fig. 7: Element weight for the chars

(Bourbigot and Duquesne, 2007). Only two coatings, RWFRIC and CWFRIC gave magnesium (Mg) and aluminum (Al) reading indicating both were loaded with the elements. Interestingly, further analysis on the chars found that Mg and Al were available in the FRIC. Similarly, only FRIC chars revealed titanium (Ti) content, again offering great fire protection (Bourbigot and Duquesne, 2007), when it was relatively undetected in the coatings.

These results show effective roles of the wool fibre in the FRIC via physical obstruction against fire and possessing high temperature materials that offer better fire protection through maintaining more high temperature elements, minimizing oxidation and having optimum level of carbon in their chars.

CONCLUSION

This research studied the mechanisms of char strengthening by three types of wool fibre reinforcement in the fired intumescent coatings' char. These mechanisms were proposed after analysing the morphology images of both the FRIC coatings and chars by SEM. EDX analysis on the other hand confirmed that FRIC was able to maintain key elements composition for ensured fire protection to the substrates.

Four strengthening mechanisms as determined by studying the fibres roles had produce strong chars of high quality and integrity. This further enhanced fire protection properties of the intumescent coatings.

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REFERENCES

Amir, N., F. Ahmad and P.S.M. Megat-Yusoff, 2011a. Characterization of inorganic non-metallic fibre reinforced epoxy-based intumescent coatings (FRIC) and chartex 7. Eng. Trans., 6: 143-151.

Amir, N., F. Ahmad and P.S.M. Megat-Yusoff, 2011b. Study on the fibre reinforced epoxy-based intumescent coating formulations and their char characteristics. J. Applied Sci., 11: 1678-1687.

Bourbigot, S. and S. Duquesne, 2007. Fire retardant polymers: Recent developments and opportunities. J. Mater. Chem., 17: 2283-2300.

Gu, J.W., G.C. Zhanga, S.L. Donga, Q.Y. Zhanga and J. Konga, 2007. Study on preparation and fire-retardant mechanism analysis of intumescent flame-retardant coatings. Surf. Coat. Technol., 201: 7835-7841.

Hanafin, J.W. and D.C. Bertrand, 2000. Low Density, Light Weight Intumescent Coating. Patent Application Publication, USA.

Jimenez, M., S. Duquesne and S. Bourbigot, 2006. Characterization of the performance of an intumescent fire protective coating. J. Surf. Coat. Technol., 201: 979-987.

Kandola, B.K., A.R. Horrocks and S. Horrocks, 2001. Complex char formation in flame-retarded fibre-intumescent combinations. Part V. exploring different Fibre/intumescent combinations. Fire Mater., 25: 153-160.

Kandola, B.K., M.H. Akonda and A.R. Horrocks, 2005. Use of high-performance fibres and intumescents as char promoters in glass-reinforced polyester composites. Poly. Degradat. Stab., 88: 123-129.

Sipaut, C.S., N. Ahmad, R. Adnan, I.A.B. Rahman, M.A. Bakar, J. Ismail and C.K. Chee, 2007. Properties and morphology of bulk epoxy composites filled with modified fumed silica-epoxy nanocomposites. J. Applied Sci., 7: 27-34.

Wang, J.Q. and W.K. Chow, 2005. A brief review on fire retardants for polymeric foams. J. Applied Polym. Sci., 97: 366-376.