



ТЕХНИКО-ТЕХНОЛОГИЧЕСКИЕ ИННОВАЦИИ

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INTRODUCTION TO CERAMIZABLE POLYMER COMPOSITES

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Abstract. In this paper ceramization (ceramification) of polymer composites is presented as a promising method for gaining flame retardancy of the materials. Because of its passive fire protecting character, ceramization effect can be applied in polymer composites, which are dedicated for work in public places like shopping centers, sport halls, galleries and museums, office buildings, theatres or cinemas and public means of transport. In case of fire, ceramizable polymer composites turn into barrier ceramic materials, ensuring integrity of objects like electrical cables, window frames, doors, ceilings, etc., exposed on flames and heat, preventing from collapsing of materials and making electricity working, enabling effective evacuation. Moreover, ceramization process decreases emission of toxic or harmful gaseous products of polymer matrix degradation as well as its smoke intensity. The paper describes the mechanisms of ceramization for various polymer composites, especially focusing on silicone rubber-based ones, basic characteristics of the materials and ways of their testing.

Key words: polymer composites, ceramization, ceramification, flame retardancy, effective chemical compounds.

The authors dedicate this work to prof. Gennady Zaikov to commemorate his 80-s anniversary.

1. Introduction

According to International Association of Fire and Rescue Service (CTIF) direct and indirect costs of fires can reach 1 % of GDP in developed countries and even up to 2 % of GDP in less-developed countries [7]. Important part of these costs is generated by fires of infrastructure, especially of buildings. Polymer materials and composites are nowadays commonly used as parts of wide range of civil engineering constructions, increasingly replacing conventional materials like concrete, wood and even metal, because of their good processability, low price, relatively high mechanical properties and corrosion resistance. However, polymer materials exhibit two major disadvantages, which are low fire resistance and low thermal stability. For example combustion heat of polyethylene or polypropylene is around 46-47 MJ/kg [28], in comparison to 32-37 MJ/kg reported for coal [27]. That's why their growing presence in building technology creates fire hazard. Moreover, lots of decorative parts and facilities are made of polymer materials, what significantly increases danger.

To eliminate this problem growing number of polymer flame retardants have been developed. But unfortunately, the most effective chemical compounds create toxic, corrosive or harmful volatile products due to radical reactions present in gas or solid phase of fire space, which can cause even more harm for people health and infrastructure than flames or heat radiation [25]. Flame retardants based on physical mechanisms of combustibility lowering, like char formation, dissipation of heat from a fire zone or volatile fuel diluting are much safer, however unfortunately also less effective. Nevertheless, due to issue of toxicity, development in field of physical flame retardants has become one of the most important way to improve fire resistance of polymer composites due to their non-toxicity. According to Morgan and Gilman [20], application of ceramic amorphous oxides of low softening point temperature as a dispersed phase in polymer composites could strongly increase properties of char being formed under fire, being one of the

most perspective way of developing new, flame retarded polymer materials. In fact, presence of this kind of glassy oxides in a composite results in possibility for creation of strong continuous ceramic phase on the surface of heat treated composite instead of rather weak and less continuous char. Thus, it begins development of new kind of passive flame retarded polymer materials – the so-called ceramizable (ceramifiable) composites.

2. Phenomenon of Ceramization

Ceramization process bases on creation of mechanically strong, porous and insulating ceramic barrier layer on the surface of composite during fire or at high temperature. This is in fact an improved char forming mechanism, but mechanical and barrier properties of ceramic phase obtained are superior to char created after heat treatment. This ceramic, protective shield prevents from diffusion of volatile fuel, created during thermal decomposition of polymer matrix, to fire zone as well as diffusion of oxygen into condensed phase of the composite, where it accelerates degradation rate of polymer macromolecules. Moreover, stiff, continuous and thermally insulating layer decreases emission of harmful, corrosive and toxic product of firing and reduces production of smoke, what can significantly increase fire safety in public use buildings, especially from the point of view of possibility for evacuation of crowds of people from places endangered by fire.

In comparison to other flame retarded polymer materials, ceramizable composites exhibit higher thermal stability, non-toxicity, excellent barrier properties and ability to sustain their shape even after complete destruction of polymer matrix at elevated temperature.

3. Organic Polymer-Based Composites

First information describing properties of ceramizable composites based on organic polymers was presented in Proceedings of "European Coatings Conference, 2006" [26]. The paper by Thomson et al. describes properties of composites based on poly(vinyl chloride) (PVC) and ethylene-propylene-diene rubber (EPDM). The authors have shown that their composites

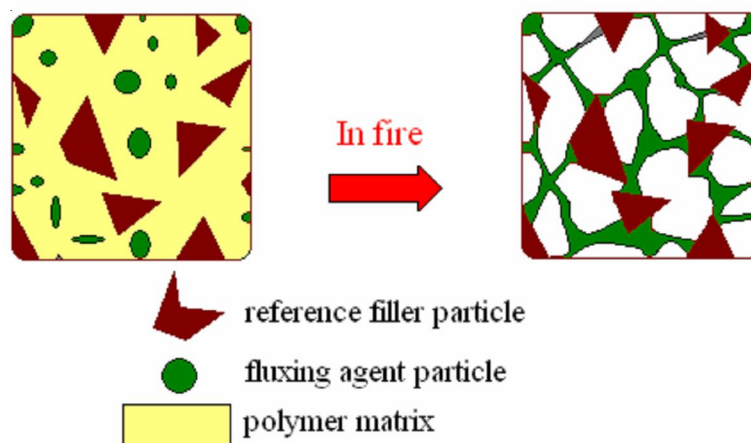


Fig. 1. Scheme of physical mechanism of ceramization involving binding action of fluxing agent particles

exhibit significantly lower heat release rate (HRR), smoke intensity and toxic carbon (II) oxide emission under cone calorimeter test. These studies have resulted in industrial application and now Australian company “Cerampolymerik” offers ceramizable PVC and EPDM composites for sale. However, this company seems to be the only manufacturer of organic polymer based ceramizable composites in the world.

In case of ceramizable composites based on organic polymers, dominating mechanism of ceramization is based on dispersed mineral phase playing the main role (Fig. 1). Because organic matrix degrades completely during heat treatment simulating fire conditions, it cannot participate in creation of ceramic phase. The most important component of dispersed phase is oxide amorphous frit called fluxing agent, which melts relatively quickly at elevated temperature and combines other, thermally stable mineral particles together, leading to formation of continuous, ceramic phase. This is physical type of ceramization mechanism, which theoretically can be applied for any kind of polymer.

Another mechanism, describing creation of continuous mineral phase on the surface of polymer composite during ceramization, is based on sintering process of refractory mineral filler particles due to condensation of hydroxyl groups present on their surface (Fig. 2). However, this process, originally proposed by Xiong et al. [30], plays minor role in forming brittle, porous ceramic phase of high endurance.

Ceramizable composites based on low density linear polyethylene (LLDPE) [29] and poly(vinyl acetate) (PVA) [1] have recently been described in scientific literature.

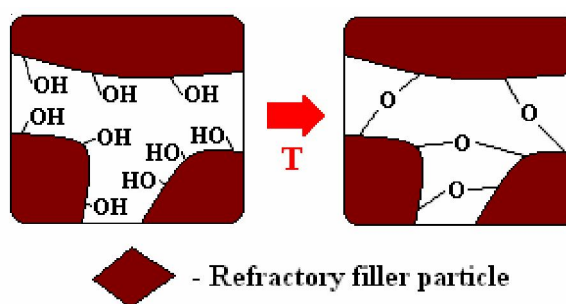


Fig. 2. Mechanism of sintering mineral filler particles during heat treatment based on condensation of hydroxyl groups

Polyethylene is one of the most representative polymer for a group of polyolefines. Currently, composites based on this material are increasingly used for cable covers production, replacing plasticized PVC, because of significantly lower toxicity level of their thermal degradation products. According to Wang et al. [29], ceramizable LLDPE composites exhibit good thermal stability and low flammability as well as high resistance against thermal shock, what make them perspective materials for cable insulations. Polyolefine-based ceramizable composites are likely to replace in cable industry commonly used more expensive silicone rubber-based composites which are much expensive.

According to Al-Hassany et al. [1], PVA based ceramizable composites can be used for sealing applications. The authors studied thermal stability and morphology of ceramizable composites after heat treatment. They have shown that composite containing kaolin particles as refractory filler exhibits better mechanical properties after ceramization than composite filled with talc particles. However, composite containing

talc exhibits better thermal stability, probably due to high shape factor of talc plates. In both cases PVA based composites sustain their shape after heat treatment, which is one of the most important factors from the point of view of fire protection because ceramic phase being created, maintains barrier properties of seal preventing from oxygen diffusion to fire zone, what eventually results in extinction of fire.

According to Zhang et al. [31], polystyrene (PS) composites containing organically modified montmorillonite clays (OMMT) are able to create porous, ceramic residue, being composed of two different layered phases after heat treatment. This residue has thin “skin” layer containing a lot of small pores and thick “cellular” layer with relatively big pores (Fig. 3). We have obtained analogical structures after heat treatment of PVC ceramizable composites, prepared in our laboratory (Fig. 4). This result proves on ceramization process being a developed version of char forming flame retardancy mechanism, acting similarly under fire conditions.

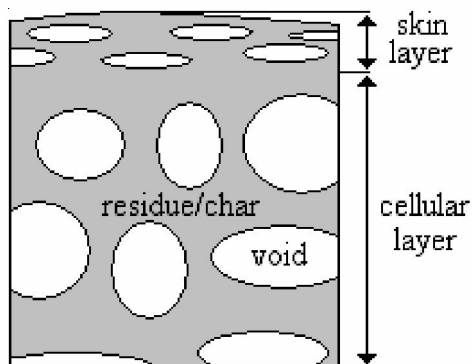


Fig. 3. Scheme of porous structure of residue obtained after heat treatment of PS/OMMT composite

4. Silicone Rubber-Based Composites

Despite a lot of research and development going on organic polymer-based composites, ceramizable composites based on silicone rubber still remain the most popular materials for cable industry. This is simply because of their ability to protect copper wire from heat melting and to sustain cable wires, maintaining integrity of electrical circuits even up to 120 min in fire. During this time all important installation, like fire sprinklers, lamps, elevators, alarm systems etc. are able to work, increasing chance for evacuation people from dangerous zones. Polysiloxanes are the best kind of polymers which can be used as a continuous phase for ceramizable composites because they are able to create amorphous silica during thermal degradation under oxidizing atmosphere. Unfilled silicone degrades creating volatile cyclosiloxanes according to three mechanisms: unzipping reaction (Fig. 5), random scission reaction (Fig. 6) and externally catalyzed reaction (Fig. 7) [11]. After depolymerization, volatile cyclosiloxanes undergo burning producing silica, which can take part in creation of ceramic phase on the surface of composite.

Unzipping reactions take place when silicone macromolecules are terminated with a polar group like hydroxyl one (-OH). These kinds of reactive groups are present in room temperature vulcanizable silicones (RTV). They allow macromolecules to create cross-linked structure as a result of reaction with curing agents, eg. silanes.

Random scission reaction dominates when silicone macromolecules are terminated with non-polar groups like methyl (-CH₃) or vinyl (-CH=CH₂) ones. These kinds of chains are characteristic for high temperature vulcanizable silicones (HTV),

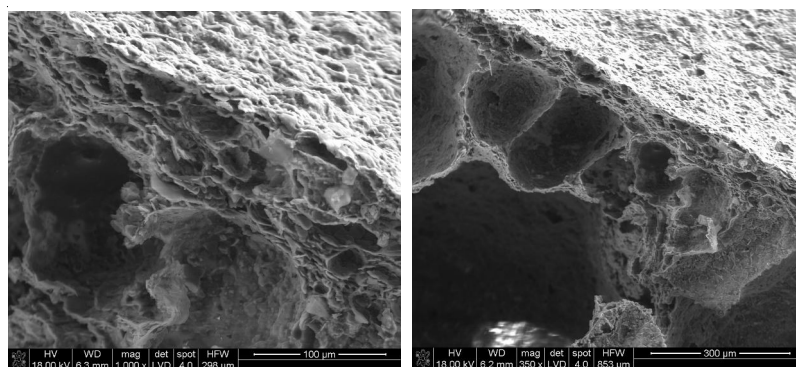


Fig. 4. SEM pictures of two layered morphology of PVC ceramizable composite subjected to heat treatment

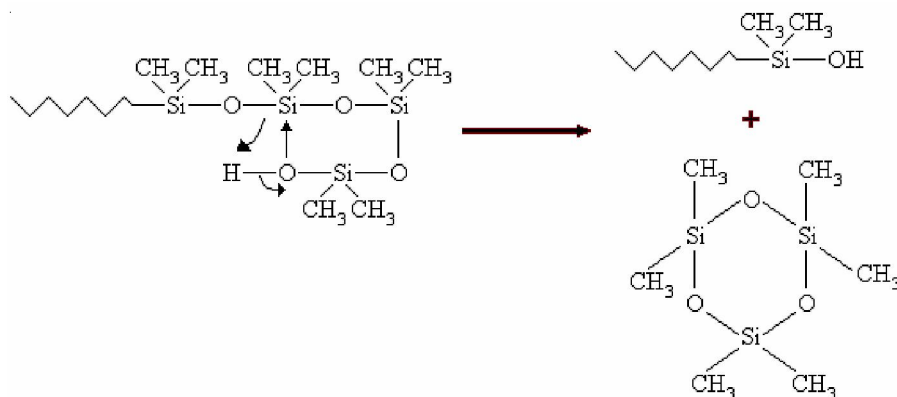


Fig. 5. Scheme of unzipping reaction mechanism of hydroxyl-terminated PDMS macromolecule

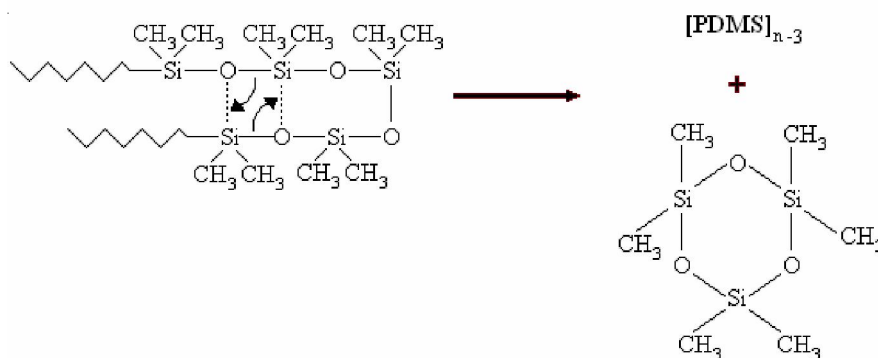


Fig. 6. Scheme of random scission reaction mechanism of PDMS chain

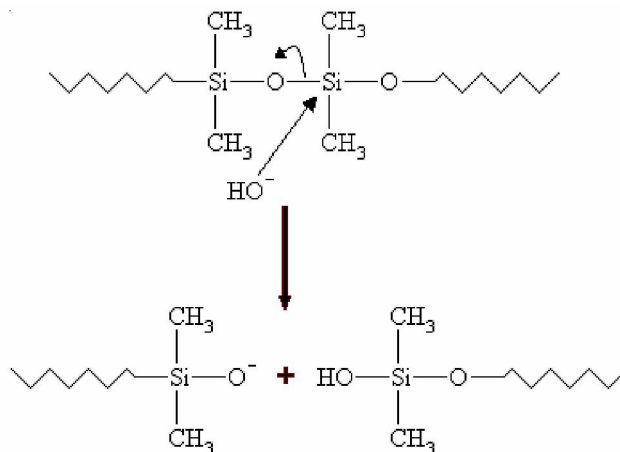


Fig. 7. Scheme of externally catalyzed degradation of PDMS macromolecule

where cross-linking of polymer bases on radical reactions with peroxides or curing reactions involving platinum catalysts.

Impurities, which exhibit ion character can also take part in thermal degradation of silicones. For example hydroxyl anions present in moisture are able to catalyzed breaking of polysiloxane chain.

According to Camino and Lomakin [8; 9], thermal degradation of silicones occur with

competition between molecular mechanisms leading to creation of cyclic volatiles (described previously) and radical mechanism leading to cross-linking of macromolecules and creation of methane (Fig. 8).

The most important advantage of using silicone rubber as a continuous phase of ceramizable composites seems to be, even without addition of fluxing agent, dispersed mineral particles are able to

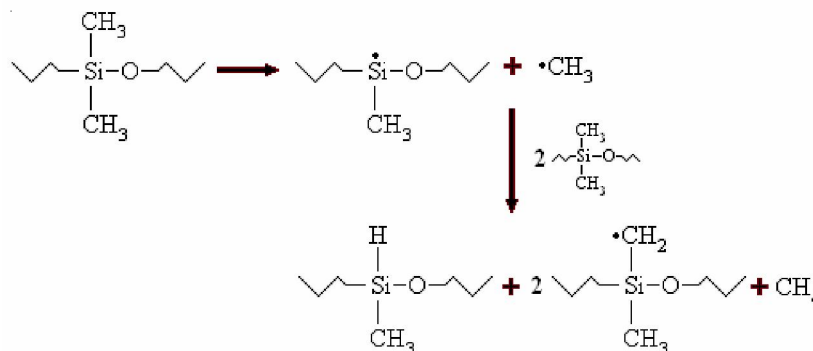


Fig. 8. Radical mechanism of thermal degradation of silicones leading to obtain cross-linked structure and methane

create ceramic continuous phase due to sintering via silica bridges created as a result of thermal degradation of silicone macromolecules bonded to their surfaces (Fig. 9) [12; 13; 17; 18; 30].

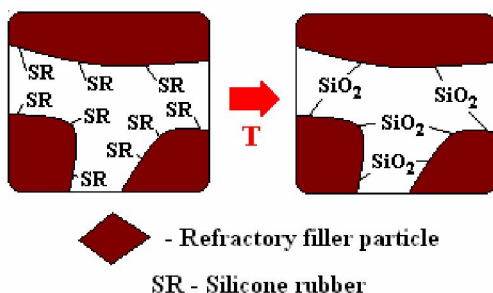


Fig. 9. Mechanism of sintering mineral filler particles during heat treatment, based on creation of silica bridges, as proposed by Xiong et al. [30]

Based on SEM analysis of ceramizable silicone composites after heat treatment, Hanu et al. [14], proposed scheme describing process of silica bridges creation on the surface of muscovite mica filler, leading to obtain continuous ceramic phase (Fig. 10). They have concluded from morphology of the system, that character of filler

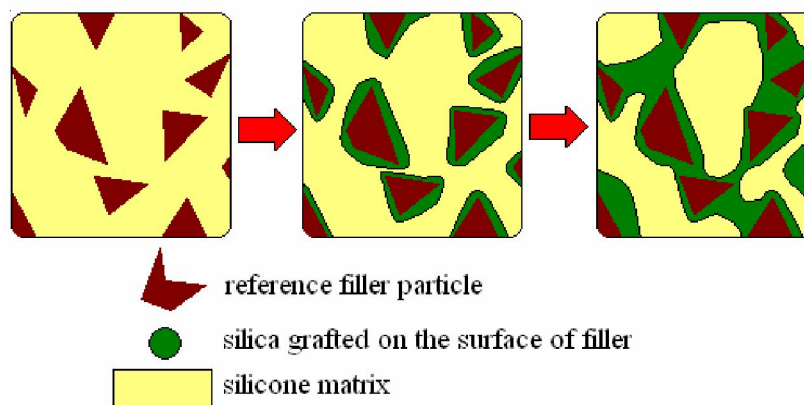


Fig. 10. Scheme of ceramic phase creation via silica bridges

surface and size of its particles are crucial factors from the point of view of ceramic shield properties.

Nevertheless, incorporation of fluxing agent into silicone rubber-based ceramizable composites significantly improves mechanical and barrier properties of ceramic phase obtained after their heat treatment [21-23]. Moreover, chemical reactions taking place between mineral particles of dispersed phase or products of their thermal oxidation, may result in creation of even more durable ceramic phase (Fig. 11). For example, calcium oxide (II) created after decarboxylation of calcium carbonate filler can react with silica obtained after thermal degradation of silicone rubber, what leads to creation of wollastonite or larnite particles.

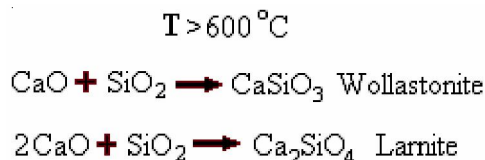


Fig. 11. Chemical reactions between mineral compounds leading to creation of more complex mineral phases

4.1. Preparation and Processing

Simple way of preparation of ceramizable polymer composites is their huge advantage. Elastomer-based composites can be prepared using classic internal or external mixers used in rubber technology. For preparation of thermoplastic composites the best way is to apply twin-screw extruder, which also allows for final forming of product shape. Major problem faced during preparation of ceramizable composites is associated with high amount of refractory mineral fillers, what strongly increases viscosity of compositions, adversely affecting their processing and forming.

Due to high amount of different mineral fillers, rheological properties of composites could be very different, even if the same polymer plays role of matrix (Fig. 12). In our previous study [4] we have compared rheological properties of various commercially available silicone rubber-based composites.

Curing of ceramizable composite mixes can be also quite challenging. Strong acidic or alkaline character of fillers can negatively influence efficiency of cross-linking, what could lead even to prevent creation of chemical bonds between macromolecules of elastomer matrix, what especially concerns peroxide cross-linking reactions. However, our recent work have shown, that high addition of alkaline mineral fillers, like MgO, CaO, or organofilized montmorillonite, can solve the curing problem of silicone composites [2].

4.2. Properties and Testing

High amount of mineral fillers strongly influences mechanical properties of ceramizable polymer composites. Their viscosity becomes significantly higher and their stiffness increases, what altogether may lead to lower impact resistance of the materials. Of course, it is possible to test ceramizable composites as any other kind of polymer materials, determining their rheological, mechanical and tribological properties, etc., but the most important types of studies are related to morphology of ceramic phase being created, its thermal properties and fire resistance of composites (heat insulation and thermal stability, respectively).

For studying thermal properties of composites thermogravimetry combined with differential scanning calorimetry (TG-DSC) can be applied. Another valuable source of information is cone calorimetry analysis, which can provide data on heat release rate (HRR) in function of temperature or time, smoke intensity or concentration of toxic products of polymer matrix degradation. In our previous work [28], we performed cone calorimetry studies for some commercially available silicone rubber-based composites

For information on flame retardancy oxygen index (OI) or limited oxygen index (LOI) is commonly determined. No less popular is vertical burning test described by UL-94 standard.

For ceramizable electrical wire covers a number of standards have been exclusively prepared, for example EN 50264-1 and EN 50382-1 – defining requirements for cables destined for railway

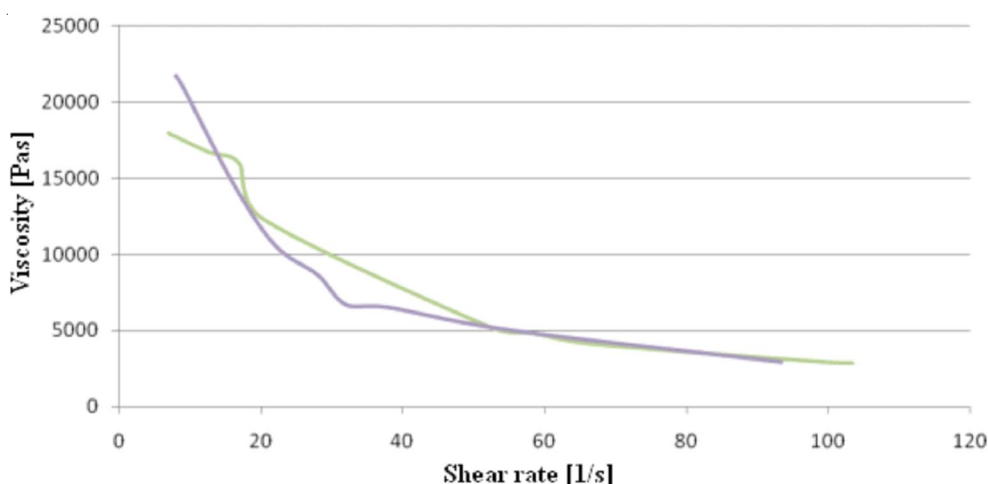


Fig. 12. Viscosity in function of shear rate of silicone rubber-based ceramizable composites originated from various producers

applications or EN 60332-1-2 describing method of fire testing for cables (Fig. 13).

In our previous study, we have described that Fourier transform infrared spectroscopy (FT-IR) of smoke and volatiles produced during ceremization process of composites can be also concern as very accurate tool for analyzing thermal properties of ceramizable composites [2].

A new challenge is to prepare suitable methodology for testing properties of ceramic phase created after composite heat/flame treatment. For qualitative analysis of its morphology scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM-EDS) seems to be sufficient. More difficult are analysis of thermal or electrical conductivity or even mechanical properties of created ceramic phase, because of sample deformation accompanying ceramization, making practically impossible to maintain required specimen shape. However, indirect information of this kind of properties can be evaluated from porosimetry analysis or simple mechanical determinations like three point bending test of ceramic phase resulted from ceramization.

Porosimetric characteristics of various commercially available ceramizable silicone

rubber-based composites [21] point on possibility to meet engineering requirements for ceramized phase by controlling morphology of materials, being related to composition, processing and anticipated thermal conditions during fire (temperature and heating rate).

5. Future Directions

Future studies will probably lead to obtain new kinds of glassy amorphous frits, acting as fluxing agents, which could provide more and more effective ceramization process of composites. Nowadays, this special kind of fillers exhibit insufficient low value of softening point temperature and additionally size of their particles still remains far too big. Moreover, very often they exhibit quite high hygroscopicity, which disturbs processing of composites. First studies describing co-milling of fluxing agent with refractory mineral fillers before compounding process are promising [30]. This is one of potential ways to decrease average size of primary particles of fluxing agent as well as to obtain its better dispersion and distribution in polymer matrix.

Another direction for development of ceramizable polymer composites will surely originate from application of various organic

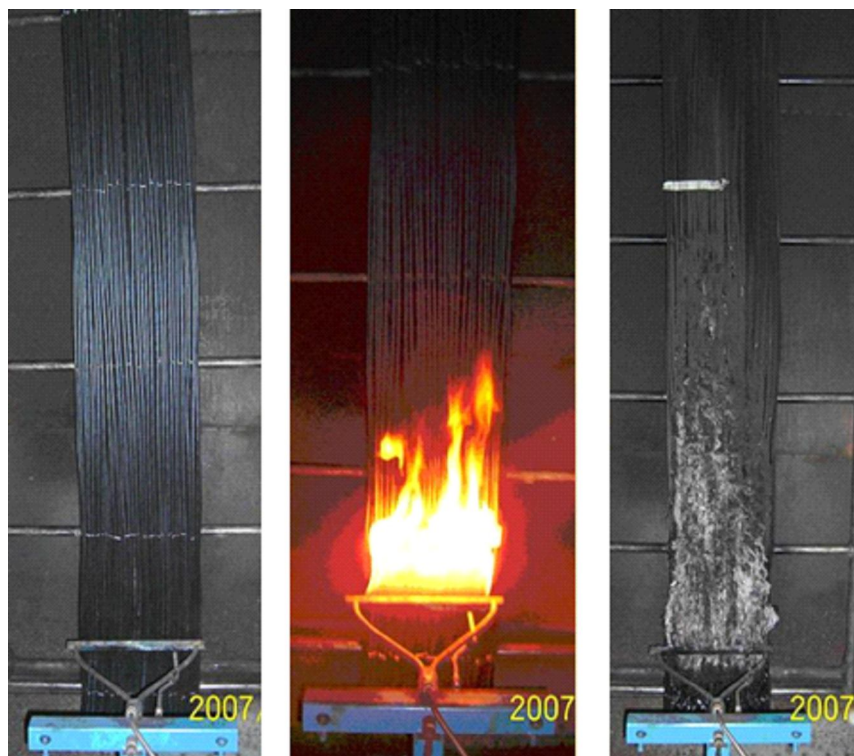


Fig. 13. Pictures representing cable test under fire conditions, according to EN 60332-1-2 standard

polymers as a composite matrix. Wide range of polymers or copolymers can be used due to their different fields of application. Already only civil engineering presents an enormous potential market. Even commonly used so far polymers, like e.g. PVC, which generate toxic or harmful products under their thermal degradation or characterizing themselves by high smoke intensity can be considered as matrices for ceramizable composites.

Another very important market seems to be cable industry, requiring permanent progress in technology and properties of silicone rubber-based composites destined for wire covers. Our recent studies have shown, that rather low mechanical properties of produced so far silicone ceremizable composites can be increased by incorporation of mineral or polyamide fibers [31].

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НОВЫЕ ВОЗМОЖНОСТИ КЕРАМИЧЕСКИХ ПОЛИМЕРНЫХ КОМПОЗИТОВ

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Аннотация. В данной работе рассматриваются керамические полимерные композиты как один из перспективных материалов для обеспечения огнестойкости. Из-за его пассивной противопожарной защиты, керамический эффект может быть применен в области полимерных композиционных материалов, которые предназначены для работы в общественных местах, таких как торговые центры, спортивные залы, галереи и музеи, офисные здания, театры или кинотеатры и общественный транспорт.

Ключевые слова: полимерные композиты, керамизация, керамический эффект, огнестойкость, эффективные химические соединения.