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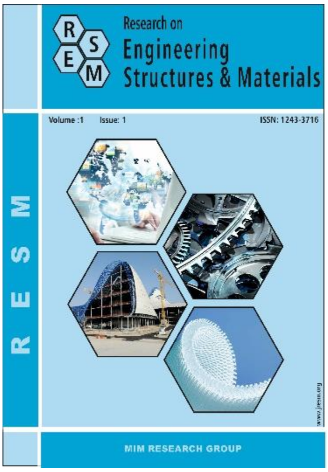

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Research Article

Multi-functional materials for military aircrafts; radar absorbing and flame retardant composites

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Abstract

Multifunctional materials with different functions can be performed simultaneously or sequentially and developed so as to improve system performance. The best means of producing multifunctional materials are composite materials with the desired properties. Composite materials can be obtained with a combination of multi-phase materials with different characteristics. Each of these phases has the ability to demonstrate a significant portion of the unique features and does not preclude other of a phase function. In this study, an epoxy resin was converted into a multi-functional material as a coating material in military aircrafts. It has the ability to perform multiple functions intended to have radar absorbing and flame retardant properties. Epoxy resin was the matrix of the composite, and huntite/hydromagnesite mineral is added to the composite matrix to achieve flame retardancy. Barium hexaferrite particles were used for radar absorbing property. The powders were produced by sol-gel method. The additive material is determined whether amount-dependent properties of varying the amount. The resulting samples were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Also the flame retardant and radar absorbing properties were investigated. As a result, both materials contribute to fulfill their primary function. Besides, it was concluded that the materials generate beneficial effects together as synergistically

1. Introduction

The performance of metallic materials available today may be inadequate in the face of worsening working conditions. Studies in this area in recent years have led to developments in the production of new materials and technology. Naturally the materials used in engineering should be able to bear the applied load to have the desired strength. In addition, the material must be well as certain surface properties to be used efficiently. Abrasion, erosion, corrosion, thermal, optical, electrical and magnetic properties of the material is related with the surface properties. Therefore, materials having desired properties can be obtained the necessary surface treatments [1,2].

The production of multifunctional materials appears as a solution to the elimination of problems in cases worsening conditions. It is required to have optimum strength, flexibility, lightness, durability, impact resistance, thermal expansion coefficient, hardness, fatigue cracking and breaking, pulling, bending strength and suitability of equivalents needs according to environmental conditions. All desired features of one metal, ceramic or polymer material is extremely difficult to find. Therefore, multifunctional composite materials attract attention in recent years. Composites, which can be defined as one of the most important of these advanced materials, are the materials consisting of two or more chemically and physically different phases (matrix phase and dispersed phase) separated

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by a distinct interface. Composites are produced when two or more materials or phases are used together to give a combination of properties that cannot be attained otherwise. The microstructures examined with the naked eye (macroscopic examination) it is possible to distinguish the components of the structure selected. Phase takes place in the plurality of macro-scale structure in which the conventional alloys. Abalone shell, wood, bone, and teeth are examples of naturally occurring composites. People have been making composites for many thousands of years. One early example is mud bricks. Mud can be dried out into a brick shape to give a building material. It is strong if you try to squash it, but it breaks quite easily if you try to bend it. So, it has good compressive strength, but poor tensile strength. Straw seems very strong if you try to stretch it, but you can crumple it up easily. By mixing mud and straw together it is possible to make bricks that are resistant to both squeezing and tearing and make excellent building blocks. [3,4]. Today they are used widely in aircraft, rockets, missiles body, high-quality sports equipment etc. The most used material in this area rubber has a place in areas that really matter the height of the costs, such as artificial bone, automotive industry, white goods, pressure-resistant pipe and they function in a wide spectrum such as marine vehicle body [5,6].

In this study, radar absorbing and flame retardant polymer composites were produced. Radar is one of the most important technological developments resulting from World War II. This technology revolutionized air and naval warfare. This is an object detection system that uses electromagnetic waves to identify the range, altitude, direction, or speed of both moving and fixed objects such as aircraft, ships, motor vehicles, weather formations, and terrain. The term RADAR was coined in 1940 by the U.S. Navy as an acronym for: Radio, Detection, And Ranging. The basic idea behind radar is very simple: a signal is transmitted, it bounces off an object and it is later received by some type of receiver (Fig. 1). This is like the type of thing that happens when sound echo's off a wall. However, radars do not use sound as a signal. Instead they use certain kinds of electromagnetic waves called radio waves and microwaves. Satellites can use radars to work on projects outside of the Earth's atmosphere and on other planets, as sound waves and ocean waves require matter to transport energy but electromagnetic waves can do so without the presence of matter. The other useful thing about electromagnetic waves is that they travel at a constant speed through a vacuum called the speed of light. Some useful information is calculated from this energy such as the time taken for it to be received, the strength of the returned signal, or the change in frequency of the signal. This information is then translated to reveal useful data: an image, a position or the velocity of your speeding car [7-9].

The amount of reflection and refraction depends on the properties of the surface and the properties of the matter which the wave was originally traveling through. This is what happens to radar signals when they hit objects. So materials structure comes into prominence whether the material reflects the signal or absorbs. In this manner, Radar Absorbing Materials (RAM) have been arised. Those materials are specially designed and shaped to absorb incident radar signals as effectively as possible and from as many incident directions as possible. The earliest forms of radar absorbing materials were the materials called Sumpf and Schornsteinfeger, a coating used by Germans during the World War II for the snorkels (or periscopes) of submarines, to lower their reflectivity in the 20-cm radar band the Allies used. The material had a layered structure and was based on graphite particles and other semi conductive materials embedded in a rubber matrix (Fig. 2) [7-9].

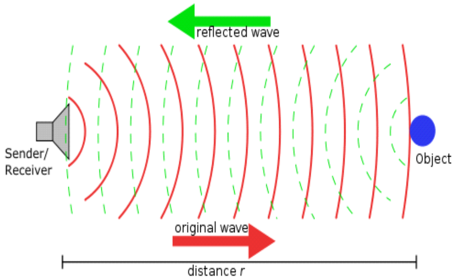

Fig. 1 Radar absorbing mechanism [10]

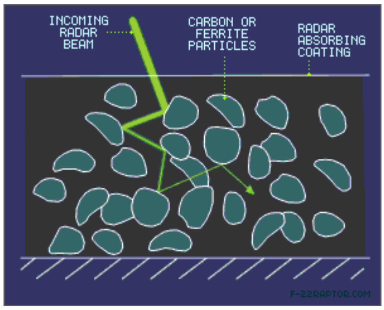

Fig. 2 Incoming and reflected radar beams [11]

Barker et al. [12] studied on absorbing microwave radiation comprising placing a microwave absorbing structure in the path of the microwave radiation. The structure comprised an array of metal plates supported over a metal substrate by vertical conducting vias, wherein the vertical conducting vias are adjustable, to increase or decrease the height of the metal plates. Saib et al. [13] presented a shielding and absorbing composite based on carbon nanotubes (CNTs) dispersed inside a polymer dielectric material. Generally, radar absorbing materials are produced by altering dielectric and magnetic properties of existing materials. The dielectric properties of a material are categorized as its permittivity and the magnetic properties as its permeability. Common dielectric materials used for absorbers, such as foams, plastics, and elastomers, have no magnetic properties. Magnetic materials, such as ferrites, iron, and cobalt nickel alloys, are used to alter the permeability of the base materials. Materials having high dielectric constant, such as carbon, graphite, and metal flakes, are used to modify the dielectric properties [7-9,14]. On the basis of carbon fibers, various studies can be seen with regard to producing radar absorbing materials. In addition, nano-Ni-Zn ferrite materials are used to absorb radar signal. Experiments performed in a carbon fiber surfaces coated nickel, graphite have also covered with a thin nickel layer [9]. Lee and his colleagues [15] have produced multilayer epoxy glass composites containing nanotubes. They carried out the result of operations in the characterization of the samples, and they stated that the optimum level of reflection loss has been determined and it can be used as radar absorbing materials. Oh and his colleagues [16] used glass containing carbon black for military purposes, they produced epoxy composites identified the radar extinguishing characteristics.

Another feature investigated in this study is flame retardant materials which can be designed to resist burning and withstand heat, or to burn slowly. Although many of materials will burn, some are naturally more resistant to fire than others. Those that are more flammable can have their fire resistance drastically improved by treatment with fire-retardant chemicals or materials. Several products are used in many applications. They can be used either alone or in combination with other flame retardants. Among the flame retardant materials used in the various markets, two main categories can be seen; halogenated and halogen-free flame retardants. Halogen containing flame retardants act in the gas phase and contribute to incompletely burned substances like black smoke and toxic CO. In a real fire many toxic gases are found. The most serious one is carbon monoxide, CO, it is a highly toxic and nonirritating gas. As CO blocks the oxygen transport of the blood, it can disturb the respiration process immediately. Even though traditional solutions based on halogens have some advantages like low loadings and good retention of mechanical properties, they have also disadvantages compared to mineral flame retardants. Fig. 3 and Fig. 4 depicts a comparison of flame retardant materials [17].

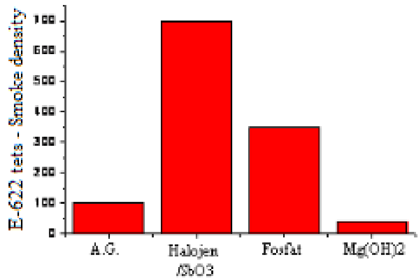

Fig. 3 Smoke formation of different flame retardants [17]

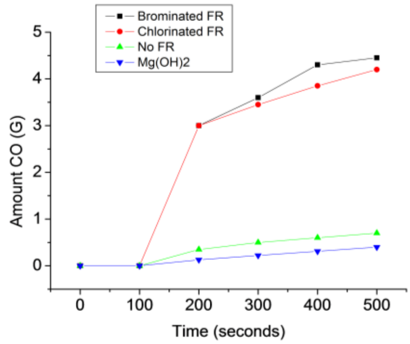

Fig. 4 CO formation of flame retardant compounds with different flame retardants [17]

There are two different products on the market as halogen-free flame-retardant materials. The first one, aluminum hydroxide, as is known ATH ($Al(OH)_3$). Other non-combustible material is magnesium hydroxide which has higher the decomposition temperature than ATH (ATH-200 °C), 340 °C. Magnesium hydroxide can be used in the plastic processing providing high temperature applications due to this advantage of [18]. Georgiades and his group [19] have provided in-situ polymerization of styrene on the high-speed mixer magnesium hydroxide. The homogeneous distribution of their products, reported that show good wetting properties and flame retardancy. Sohu and colleagues [20] have sought to improve in terms of flame retardant properties by the addition of methyl phosphonates. Flame retardant properties have improved dramatically and provides incredible fall while the glass transition temperature.

Halogen-free inorganic minerals have hydroxides. These structures decompose in between 200 °C and 400 °C and endothermic degradation of carbon dioxide and water vapor comes to light. A kind of cooling effect on the reaction and the impact of the inert gas atmosphere is composed with a similar burning surface layer formed in the ceramic structure. This layer protects against forward movement of a burning combustible surface and temperature. Those flame-retarding polymer composites by intumescence are essentially a special case of a condensed phase activity without apparent involvement of radical trap mechanisms in the gaseous phase. Intumescence involves an increase in volume of the burning substrate as a result of network or char formation. For ingress of oxygen to the fuel, this char serves as a barrier and also as a medium in which heat can be dissipated (Fig. 5) [21].

In this study, to obtain radar signal absorbing, barium ferrite powders were used, and for fire retardant property huntite and hydromagnesite mineral was utilized. Barium hexaferrite is widely used due to its high stability, excellent high frequency response, narrow switching field distribution, and the temperature coefficient of the coercivity in various applications. Barium ferrite with hexagonal molecular structure has fairly large magneto crystalline anisotropy, high curie temperature, and relatively large magnetization as well as chemical stability and corrosion stability, its chemical formula is $BaFe_{12}O_{19}$ [22,23]. Huntite and hydromagnesite mineral was introduced to the market in the late 1980s, as one of the magnesium containing sources for mineral flame retardants. The deposit normally consists of physical blends of two minerals huntite and hydromagnesite with varying ratios in between 30% and 40% huntite and 60 and 70% hydromagnesite

The level of impurities is very low, the most important ones are other white carbonate minerals such as aragonite, calcite, and dolomite. Physical densities of huntite are 2.7 g/cm^3 and 2.24 g/cm^3, respectively. Environmentally friendly halogen-free flame retardant substances huntite chemical formula is $Mg_3Ca(CO_3)_4$ and Hydromagnesite formula is $Mg_4(CO_3)_3(OH)_2.3H_2O$. Non-corrosive minerals, both effective smoke suppressant in many plastics with special finishes are able to find appropriate use of space. There is also an excellent addition to smoke and acid suppression sweep feature. Huntite and hydromagnesite number of degradation reactions shown in Eqs. (1) and (2).

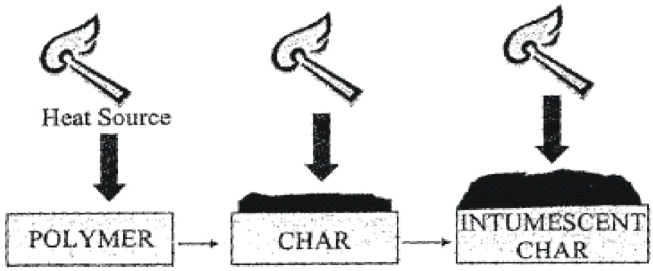

Fig. 5 Char and intumescence formation [21]

Huntite: $$Mg_3Ca(CO_3)_4 \rightarrow 3MgO + CaO + 4CO_2 \quad (1)$$
Hydromagnezite: $$Mg_4(CO_3)_3(OH)_2.3H_2O \rightarrow 4MgO + 3CO_2 + 4H_2O \quad (2)$$

In this study, radar absorbing and flame retardant composite coating were produced for military aircrafts. With this design, an epoxy resin was converted capable multiple functions of fulfilling multifunctional. Epoxy resin was used as the matrix of the composite, and huntite/hydromagnesite mineral was added to the composite matrix to achieve flame retardant property. Barium hexaferrite particles were used to radar absorbing property, produced by Sol-Gel method. The additive material is determined whether amount-dependent properties of varying the amount. Obtained samples were characterized with SEM and XRD characterization devices. Also the samples were subjected to the flame retardant and radar absorbing test, and presence of both properties were investigated.

2. Materials and Method

In the production of composite materials epoxy resin was used as a matrix material. Huntite/hydromagnesite mineral was used to achieve flame retardancy. The ore was extracted from Isparta Yalvac Tırtar village. It was crushed and ground by using jaw crusher and ball mill in Muğla Sıtkı Koçman University laboratory. After the process of sieving, the powder below 38 microns' mineral powder was selected to be used as an additive in composites production. The barium hexaferrite powder used for radar absorbing was produced by sol-gel method. Sol-gel technology is used for obtaining a high purity, controlled colloidal particle shape, size and size distribution. Low operation temperature and dilute conditions is possible in a mixture at the atomic level. Barium hexaferrite powders at atomic scale were prepared by using citrate sol–gel process. Used precursors were barium nitrate and ferric citrate mono hydrate, chelating agent was citric acid monohydrate and pH regulator was ammonium hydroxide. The solutions were vigorously mixed by magnetic stirrer until the transparent solution was obtained. Ammonium hydroxide was added until reaching of the pH value of the solution to 7 at room temperature and then mixed by magnetic stirrer. Thus it was aimed to provide homogenous suspension and stable pH condition in the solution after whole solution preparation. The solution was kept in water bath at 80 °C for 15 h. Then obtained wet gel was treated at 550 °C in Memmert oven for 6 hours for preparing dry gel. Finally, barium ferrite powders were obtained by sintering in Ankatest-1 tube oven at 1000 °C for 5 h in the Muğla Sıtkı Koçman University lab. Huntite and hydromagnesite, barium ferrite into the epoxy resin powder were mixed by adding the ratios shown in Table 1 are coated on the glass substrate with metal and composite obtained. The coatings are dried allowed to stand at room temperature for 24 hours. The obtained samples were characterized by using X-ray diffraction (XRD) and scanning electron microscopy (SEM). Moreover, as the main objective of this research radar absorbing and flame-retardant test were performed. The thicknesses of the coatings were in the range of 0.5-0.7 mm.

Tablo 1 Brium ferrite and huntite hydromagnesite amount (by weight) in the composite samples

Sample code	Huntite/hydromagnezite (%w)	Barium ferrite (%w)
H0B0	0	0
H5B1	50	10
H5B2	50	20
H6B1	60	10

3. Results and Discussion

XRD patterns of huntite/hydromagnesite and barium hexaferrite powders are shown in Fig. 6(a) and 6(b). In the mineral mixture of hydromagnesite and huntite, the essential minerals are huntite, hydromagnesite and dolomite. Chemical analysis is generally huntite (46%), hydromagnesite (46%), magnesite (4%), aragonite (3%) and calcite (1%) according to the literature researches [17,18]. For the barium ferrite powders, the main phase seems barium ferrite phase, the presence of this phase as a major phase. Besides, a little amount of iron oxide (Fe_22O_3) as a minor phase observed in the powders. Any amorphous phase is not indicated. A good agreement is achieved with Ref. [24] that the reaction was sufficient for complete conversion of the metal compounds to metal oxides. The reason for that may be enough sintering temperature 1000 °C.

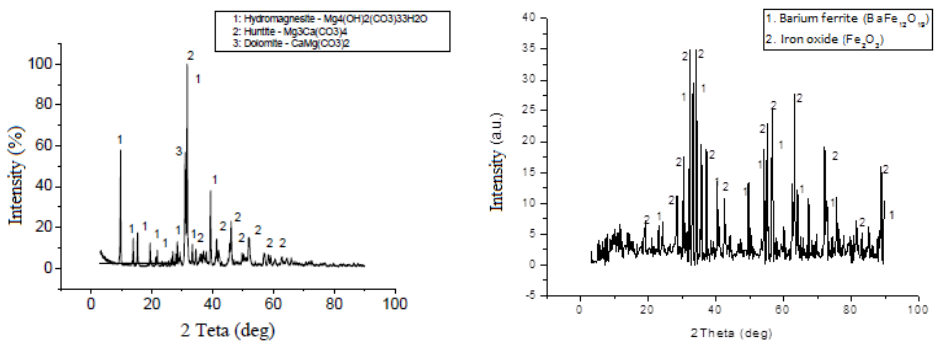

Fig. 6 XRD patterns of (a) huntite/hydromagnesite and (b) barium hexaferrite powders

SEM images of barium ferrite and huntite/hydromagnesite minerals reinforced coatings are given in Fig. 7. The additive powders can be seen from the Figures as different from pure epoxy coating. It was understood from the coatings that the powders are distributed homogeneously.

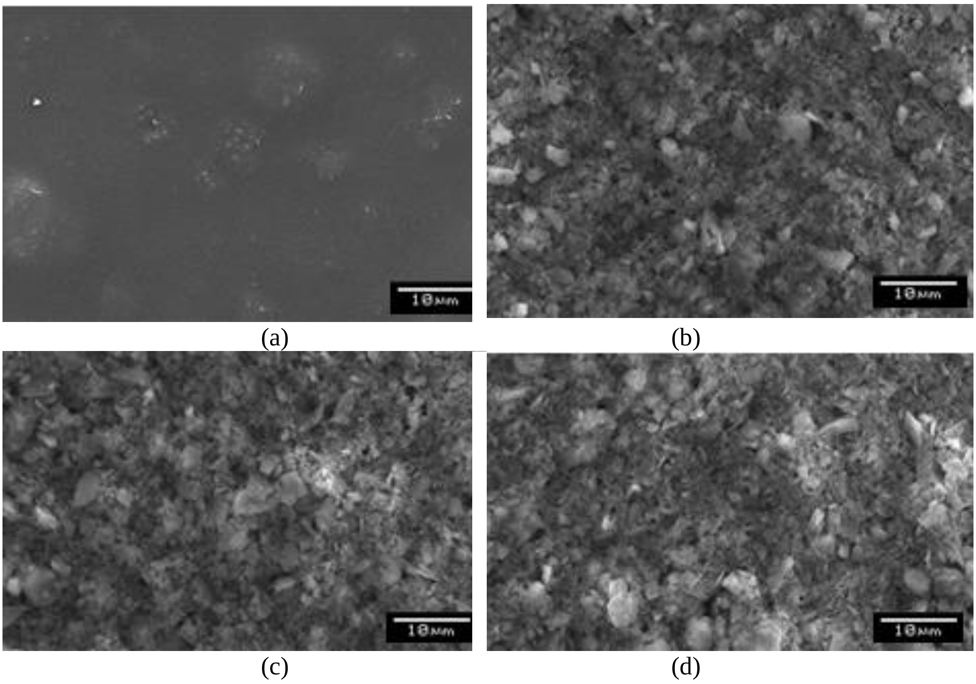

Fig. 7 SEM micrographs of coatings; (a) H0B0, (b) H5B1, (c) H5B2 and (d) H6B1

The electromagnetic parameters of the composites were measured with transmission/reflection method in the region of 8–12 GHz. with a Network Analyzer. In Fig. 8, the setup the incident power, delivered by the network analyzer and the reflected and transmitted power, measured by the network analyzer, are depicted schematically [23].

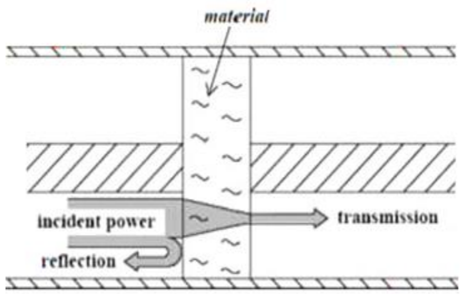

Fig. 8 Schematic illustration of transmission/reflection method.

Fig. 9 shows the microwave absorbing properties of $BaFe_{12}O_{19}$ powder reinforced epoxy coatings. The axes are showing absorbance (%) vs frequency (GHz). It means that in the

Network Analyzer, microwaves were sent to the samples, and Absorbance (%) value showing how much of those microwaves were absorbed by our samples. It can be seen that there is no any absorbance value for the sample of H0B0 which is pure epoxy coating. However, at a certain rate of radio wave absorbing activity has been observed in barium ferrite powder reinforced coatings. It was found out that the percentage of absorption increased by increasing additive content in the composite coatings [25]. The highest absorbance value was achieved at the sample of H5B2 as 8.56%. The reason for that is the large magneto crystalline anisotropy of $BaFe_{12}O_{19}$. Because of the spin orientations of the Fe^{3+} ions, the numbers of unpaired electrons are settled into their 4s and 3d shells. Those not fully filled shells lead to magnetic moment. On the other hand, the magnetic moment of barium hexaferrite particles is related with not only the unpaired electrons but also angular momentum which represents the product of the body's rotational inertia and rotational velocity. These unique characteristics enhance largely the microwave absorption performance of barium hexaferrite reinforced composites [26].

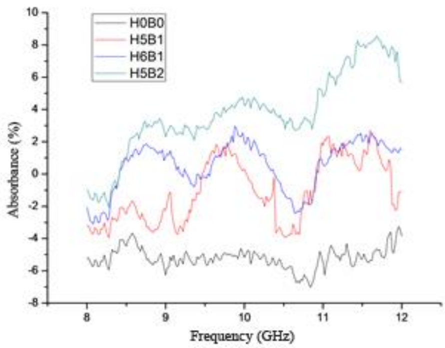

Fig. 9 Radar absorbing test results of composite coatings

Related with the flammability, UL94 test applied to the composite samples. The flame retardant test apparatus is settled up in the laboratory in Faculty of Engineering Muğla Sıtkı Koçman University accordance with the relevant testing standards. UL-94 test apparatus is shown schematically in Fig. 10 [27]. The samples were exposed to the flame in the test and the flammability of the samples were estimated according to given the standard. Table 2 shows the test results. Accordingly, comprising flame retardant property was determined in the hydromagnesite and huntite reinforced coatings, and it is stated that increasing the amount of mineral additives of the polymer sample increased the flame retarding property.

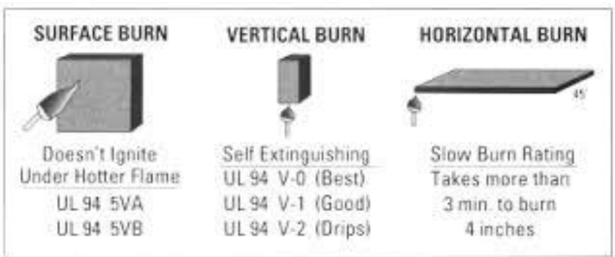

Fig. 10 Schematic illustration of UL-94 flame retardant test [21]

Table 2 UL-94 flame retardant test results of composites

Sample code	Starting flame	After flame	Result
H0B0	50 s	>180 s	Unsuccessful
H5B1	No Flame	No Flame	Successful
H5B2	No Flame	No Flame	Successful
H6B1	No Flame	No Flame	Successful

4. Conclusion

It was aimed that the multifunctional material is capable of simultaneously and intended to have radar absorbing and flame retardant properties. Barium hexaferrite particles were used to radar absorbing behaviour produced by Sol-Gel method. Huntite/hydromagnesite minerals were subjected to the crushing, grinding and screening procedures to be used as flame retardant additives. The produced composite coatings are investigated by imposing upon both flame retardant and radar absorbing tests. Accordingly, the contribution of barium ferrite powders detected radio waves absorption by approximately 9%. In the flame retardancy test it was investigated that increasing additive amount, the flammability increased. As a result, both materials contribute to fulfill their primary functions. Besides, it was concluded that the materials generate beneficial effects together as synergistically. To improve radar absorbing percentage the thickness of the coating can be increased. The work is planned to continue in this direction.

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