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Afterburner microscopic analysis of the nozzle insert material of the solid propellant rocket engine: theoretical conception (Part I)

Análisis microscópico post-quema del material del inserto de la tubería de motor-cohete a propelente sólido: concepción teórica (Parte I)

Análise microscópica pós-queima do material do inserto da tubeira de motor foguete a propelente sólido:conceituação teórica (Parte I)

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ABSTRACT

This work presents a study conducted in the Materials Division (AMR), subordinated to the Institute of Aeronautics and Space (*Instituto de Aeronáutica e Espaço* - IAE), organization of the Department of Aerospace Science and Technology (DCTA), to investigate the microstructural behavior of carbon/carbon composite based material used as heat shield/insert in nozzle throat of rockets. These systems are subjected to an intense heat flow from the gases at high speed, which lead to the ablation phenomenon in the nozzle regions in solid propulsion engines of S43 vehicles, for example. Ablation is an erosive phenomenon that occurs in regions of the thermal protection system and whose material is removed by thermomechanical, thermochemical and thermophysical or combined influences. Thus, in order to maintain the integrity of the nozzle, materials such as Thermal Protection Systems (TPS) are used. The materials for thermal protection can be classified, according to the predominant mechanism of protection, in ablatives and reirradiant. Most of the ablative materials are composites reinforced with structural fibers (silica or carbon, for example) and bonded with organic thermo rigid resins and the class of reirradiant materials include carbon mesh thermostructural composites reinforced with carbon fibers (CRFC), composites with silicon carbon/silicon carbide (C/SiC) hybrid mesh, and the silicon carbide mesh and fibers composites (SiC/SiC) and covalent ceramic materials such as ZrC, HfC and TaC, for example, mainly in the form of internal modifying materials or as coatings. The preparation of the samples and concepts related to the materials are presented as part I of the work and the assessment of the microstructural behavior of the S43 engine will be performed by stereomicroscopy, Optical Microscopy (MO) and Scanning Electron Microscopy (SEM), as part II.

Keywords: Nozzle insert. Rocket engine. Solid propulsion. CRFC insert.

RESUMEN

El presente trabajo presenta un estudio conducido en la División de Materiales (AMR), subordinada al Instituto de Aeronáutica y Espacio (IAE), organización del Departamento de Ciencia y Tecnología Aeroespacial (DCTA), para investigar el comportamiento microestructural del material a base de compuesto carbono/carbón utilizado como protección térmica/inserto en la garganta de los tubos de cohete. Estos sistemas se someten a un flujo intenso de calor proveniente de los gases de alta velocidad, que llevan

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The acronyms and abbreviations contained in this article correspond to the ones used in the original article in Portuguese.

al fenómeno de ablación en las regiones de la tubería en motores a propulsión sólida de vehículos S43, por ejemplo. La ablación es un fenómeno erosivo que ocurre en regiones del sistema de protección térmica y cuyo material es removido por influencias termomecánicas, termoquímicas y termofísicas o combinadas. Así, para mantener la integridad de la tobera, se utilizan materiales como Sistemas de Protección Térmica (SPT). Los materiales para protección térmica pueden clasificarse, según el mecanismo predominante de protección, en ablativos y reirradiantes. La mayoría de los materiales ablativos son compuestos reforzados con fibras estructurales (sílice o carbono, por ejemplo) y unidos con resinas termorregidas orgánicas y, en la clase de materiales reirradiantes, se encuentran los composites termoestructurales con matriz de carbono, reforzados con fibras de carbono (CRFC), compuestos con matriz híbrida de carbono/carburo de silicio (C/SiC), y los compuestos de matriz y fibras de carburo de silicio (SiC/SiC) y los materiales cerámicos covalentes, como ZrC, HfC y TaC, por ejemplo , principalmente en forma de materiales modificadores internos o como recubrimientos. La preparación de las muestras y concepciones inherentes a los materiales se presenta como parte I del trabajo y la evaluación del comportamiento microestructural del motor S43 será realizada por estéreo microscopia, Microscopia Óptica (MO) y Microscopia Electrónica de Barrido (MEV), será presentada, en trabajo futuro, como parte II.

Palabras clave: *Inserto de la tobera. Motor-cohete. Propulsión sólida. Inserto de CRFC.*

RESUMO

O presente trabalho apresenta estudo conduzido na Divisão de Materiais (AMR), subordinada ao Instituto de Aeronáutica e Espaço (IAE), organização do Departamento de Ciência e Tecnologia Aeroespacial (DCTA), para investigar o comportamento microestrutural do material a base de compósito carbono/carbono usado como proteção térmica/inserto em garganta de tubeira de foguetes. Esses sistemas são submetidos a um fluxo intenso de calor proveniente dos gases em alta velocidade, que levam ao fenômeno de ablação nas regiões da tubeira em motores a propulsão sólida de veículos S43, por exemplo. A ablação é um fenômeno erosivo que ocorre em regiões do sistema de proteção térmica e cujo material é removido por influências termomecânicas, termoquímicas e termofísicas ou combinadas. Assim, para manter a integridade da tubeira, utilizam-se materiais como Sistemas de Proteção Térmica (SPT). Os materiais para proteção térmica podem ser classificados, conforme o mecanismo predominante de proteção, em ablativos e reirradiantes. A maioria dos materiais ablativos são compósitos reforçados com fibras estruturais (sílica ou carbono, por exemplo) e unidos com resinas termorrígidas orgânicas e, na classe de materiais reirradiantes, encontram-se os compósitos termoestruturais com matriz de carbono, reforçados com fibras de carbono (CRFC), compósitos com matriz híbrida de carbono/carbeto de silício (C/SiC), e os compósitos de matriz e fibras de carbeto de silício (SiC/ SiC) e os materiais cerâmicos covalentes, como ZrC, HfC e TaC, por exemplo, principalmente na forma de materiais modificadores internos ou como recobrimentos. A preparação das amostras e conceituações inerentes aos materiais são apresentadas como parte I do trabalho e a avaliação do comportamento microestrutural do motor S43 será realizada por estéreo microscopia, Microscopia Ótica (MO) e Microscopia Eletrônica de Varredura (MEV), será apresentada, em trabalho futuro, como parte II.

Palavras-chave: *Inserto da tubeira. Motor-foguete. Propulsão sólida. Inserto de CRFC.*

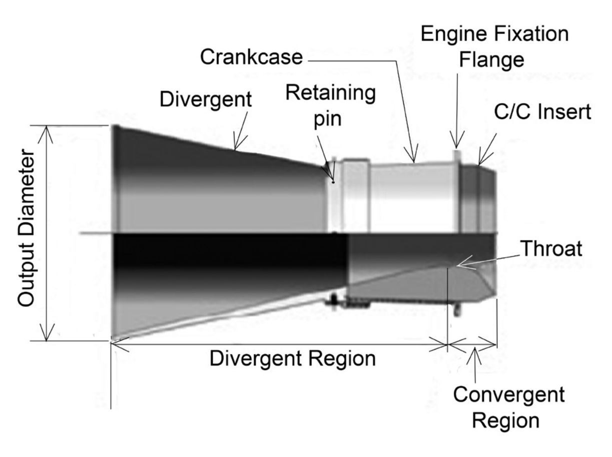
1 INTRODUCTION

The rocket engines used in satellite launch vehicles and suborbital rockets, developed at the Instituto de Aeronáutica e Espaço (IAE), have chemical propulsion systems, where there is combustion reaction of the propellant, either solid, liquid or hybrid. Thus, following the classical concept, the production of thermal energy, in the form of gases, occurs under high temperature and pressure. The gases generated in the combustion chamber are ejected by the nozzle where the thermal energy is converted into kinetic energy, resulting in thrust for the rocket propulsion (SUTTON, 1992).

1.1 Nozzle

According to Palmerio (2017: 73), the choice of materials and the design of the internal geometric profile of the nozzle are essential for the flow and expansion of the gases from the burning of the propellant to be efficient for thrust generation of the launch vehicle. It is not enough that gases are produced and released. In order to be able to induce high speed to the rocket, it is necessary to accelerate the gases produced so that they reach high speeds in the output section. In order to achieve this effect, the carbon mesh composite insert, reinforced with carbon fibers (CRFC) (Figure 1), a nozzle component, has a convergent region inside it, which initiates the acceleration process, another divergent one, in which the gases are expanded to supersonic velocity (Mach number> 1) until ejection through the output section. At the transition between the convergent and divergent regions, the critical section called throat, which has the smallest diameter and in which section the gases reach the sonic velocity (Mach number = 1). Figure 1 schematically illustrates a nozzle of a typical rocket engine used in launch vehicles for suborbital missions.

Figure 1 – Nozzle assembly of a launch vehicle.



Source: Palmerio (2017).

The crankcase, shown in Figure 1, has the following functions:

i) fixing of the nozzle to the engine envelope through a flange;

ii) receptacle of the insert with a slightly conical inner surface in order to avoid expulsion of the insert due to the passage of gases through the nozzle; and

iii) mechanical resistance to resist the internal pressure in the divergent region.

Palmerio (2017) also mentions that the region of the throat insert is exposed to an intense heat flow from the gases at high speed. To resist these conditions, materials with special characteristics that will form a thermal protection barrier are used, providing enough insulation to maintain the temperature and integrity of the metal crankcase structure and the launch vehicle as a whole.

According to Silva (2009), in general, materials of different characteristics are used in Thermal Protection Systems (TPS) in the aerospace industry with specific properties, such as low specific mass, high mechanical strength and high melting point. Each type of material is used according to its characteristics for the thermal protection of the nozzle. These materials can be classified, according to the predominant mechanism of thermal protection, in: ablatives or reirradiants.

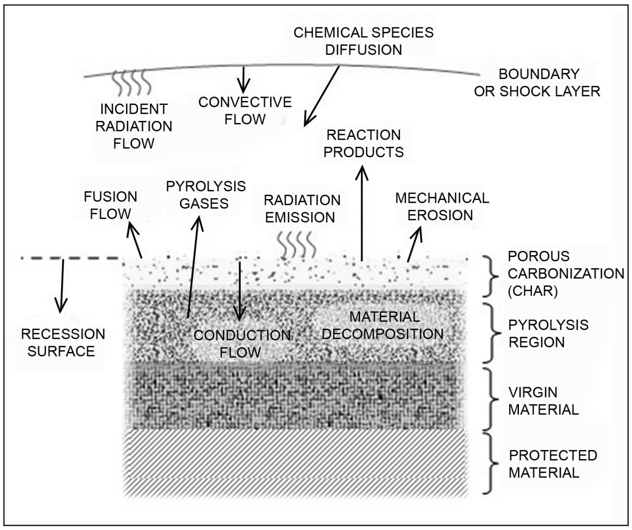
1.1.1 Ablative Materials

Ablative materials are used as a practical method to alleviate extreme aerodynamic heating conditions occurring during reentry of space systems in the atmosphere as well as in the high-velocity flow of the exhaust gases from the propellants. In this TPS the thermal energy dissipation occurs due to the loss of mass and the change of the phase of the material. Its main characteristics are high temperature thermal resistance, erosion resistance, thermal shock and impact, as well as low thermal conductivity and high specific heat (SILVA, 2009).

The TPSs that use ablative materials are the simplest and consist of the external covering of the vehicle by means of a shield with great thermal capacity to resist the intense heat flow emanated from the kinetic energy loss in ballistic trajectory in the atmospheric reentry, and whose heating rates and deceleration are very intense in a short period of time.

The predominantly ablative composites are materials reinforced with structural fibers (silica, quartz or carbon) and bonded with organic thermo rigid resins, usually phenolic resins (LAUB; VENKATAPATHY, 2003). When heated, the pyrolysis of the thermo rigid mesh (resin) occurs, which converts to porous carbon on the surface of the material and tends to create a gas layer close to that surface with a lower temperature than the temperature of the external gas from the flow of high enthalpy. The gases flow towards the hot surface and end up being injected into the boundary layer, as shown in Figure 2.

Figure 2 – Heat accommodation mechanisms of ablative materials.



Source: Pulci et al. (2010).

In the ablative process, it is considered that the initial incident energy at the surface coming from the thermal flows by radiation, convection and conduction is absorbed and then conducted into the material at a rate that depends on its thermal conductivity (PULCI et al., 2010). Thus, as soon as the heat absorption capacity of the material is exceeded, in other words, it exceeds the limits of thermal and physical stability of the material, the thermal decomposition/degradation process of the material begins, as observed in Figure 2.

This degradation involves endothermic processes that absorb much of the incident heat, preventing it from being transported to more internal regions of the material. The organic components of the material (thermo rigid resin) are then pyrolyzed at this stage, generating volatiles (pyrolysis gases) of varied composition and forming a layer of porous carbon or carbonaceous residue. In the literature the porous carbon surface formed is called char. These physicochemical changes cause alterations in the thermophysical properties of the material, causing its surface to acquire typically insulating and refractory characteristics.

The ablation mechanisms provide sufficient thermal insulation to maintain the interior of the vehicle, or system, space at a mild temperature (100 ºC), according to the gradient shown in Figure 3.

1.1.2 Reirradiant materials

In the case where the TPS is reirradiant, part of the energy absorbed from the external flow is returned to the environment in the form of radiation. This amount of energy returned can be estimated by the Stefan-Boltzmann Law, expressed by Equation 1.

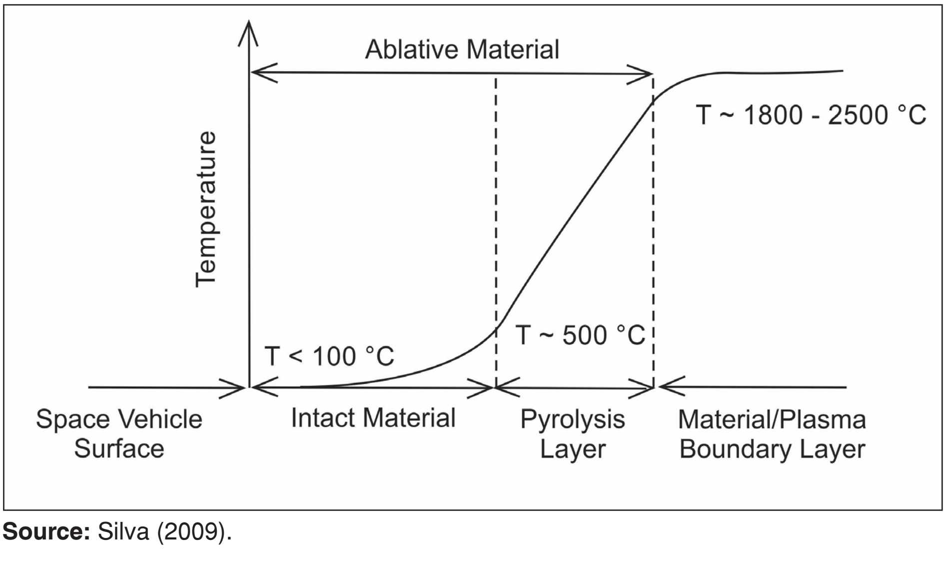
$$\dot{Q} = \sigma_{sb} \varepsilon \, T_w^4 \qquad (1)$$

Where $\dot{Q}$ represents the reirradiated energy per unit of time and per unit of area, σ_{sb} is the Stefan-Boltzmann constant 5,67032×10^{-8} W/(m^{2}K^4), ε the emissivity of the material and T_w the absolute temperature of the material (WITTMANN, 2009, p. 89).

Materials used in reirradiant TPSs have the characteristic of low erosion wear, given the incident flow conditions. These materials are used in reusable thermal protection systems.

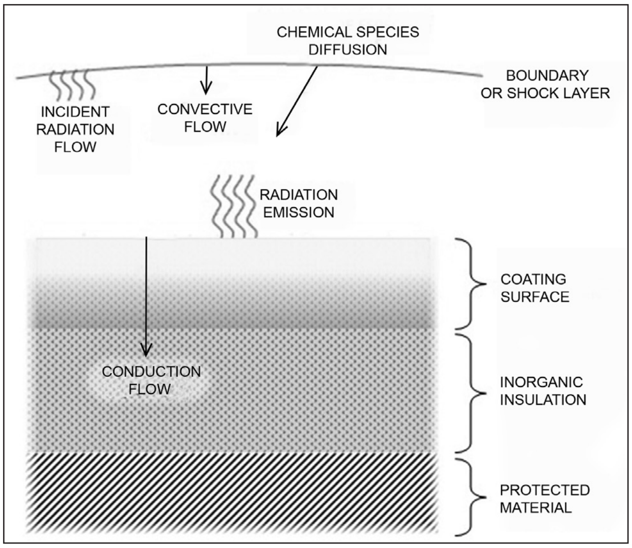
According to Figure 4, in a reirradiant material, the resulting radiative and convective heat flow will be significantly reirradiated by the heated coating surface, the rest being conducted into the material (a simpler mechanism than that of the essentially ablative materials). The advantage of this system is the possibility of reuse, since its high emissivity, which maximizes the amount of reirradiated energy, and the low surface catalysis that minimizes the convective heating, lead to the suppression and recombination of the dissociated species in the boundary layer with heated surface. Another advantage of this type of material is that the primary insulation (usually inorganic) has a low thermal conductivity, which minimizes the mass of material needed to isolate the protected structure (LAUB; VENKATAPATHY, 2003).

Figure 3 - Temperature gradient according to the layers of an ablative material.



Source: Silva (2009).

Figure 4 – Mechanisms of heat accommodation of reirradiant materials (reusable).



Source: Laub and Venkatapathy (2003).

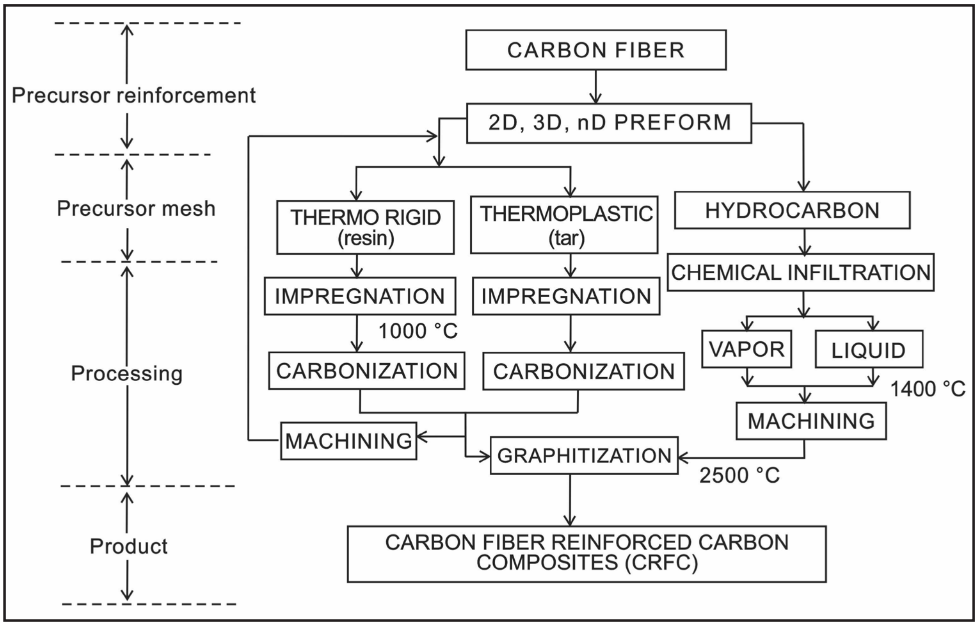
This class of materials includes carbon fiber reinforced carbon composites (CRFCs), composites with carbon/silicon carbide hybrid mesh (C/SiC), and mesh composites and silicon carbide fibers composites (SiC/SiC) and covalent ceramic materials, such as ZrC, HfC and TaC, for example mainly in the form of internal modifiers or in the form of coatings (LIUYANG; XING; YIGUANG, 2017; SILVA; PARDINI; BITTENCOURT, 2016; THIYAGARAJAN, 1996; YONG-JIE et al, 2013). These materials usually have high emissivity ($\varepsilon > 0.8$), allowing to protect structures for long period of time.

Like other space vehicles structures, TPSs are obtained with geometry suitable for use of the component, for example the launch vehicle nozzle throat material, as shown in Figure 1, whose thermal protections are located in the output region of the gases generated by burning propellants (nozzle throat).

In item 1.1 it was verified that the region of the throat is submitted to the intense heat of the gases originating from the burning of the propellant that moves at high speed. The gas and particulate flow from the propellant combustion associated with the generation and propagation of heat by the structure of the throat insert can affect the mechanical and thermal properties of the throat. To resist these conditions, reirradiant TPSs obtained by multi-directional (nD) carbon fiber reinforced carbon (CRFC) composites are used.

In the case of CRFC composites, for example, the production processes may be either by liquid phase or gas phase processes. The diagram shown in Figure 5 schematically presents the processing routes of these materials. It can be seen in Figure 5 that the CRFC composites are used by a combination of the precursor reinforcement consisting of carbon fibers, arranged in the form of preforms (nD), with the precursor carbon meshes, which may be derived from thermo rigid polymers (resin), thermoplastics (tars) and hydrocarbon gases. The processing routes, mesh precursors and the reinforcement are defined based on the final properties and desired geometries of the component to be obtained. In Figure 5, it can be seen that both the processing route of CRFC

Figure 5 – Simplified schematic diagram of the processing steps of carbon fiber reinforced carbon composites.



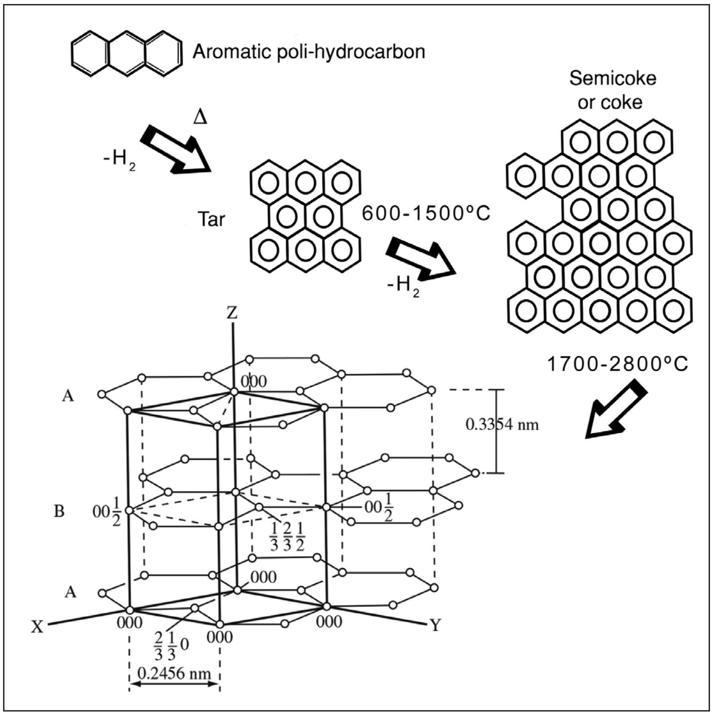
Source: Pardini and Gonçalves (2009).

composites based on thermo rigid resins and the route using thermoplastic meshes (tars) are liquid phase impregnation processes, in which the porous substrate (preform) is formed of carbon fibers (FITZER; MANOCHA, 1988; GONÇALVES, 2008). Finally, there is the route, whose impregnation occurs by means of hydrocarbon gas, which contains carbon in its molecule, an element that is decomposed in the porous substrate of carbon fibers. In this case, the process of obtaining the composite is called chemical infiltration in the gas phase (CVI).

In the thermo rigid liquid phase process, the precursor mesh consists of thermo rigid resins, which cure (polymerize) at low temperatures (T <250°C) and are converted to a carbonaceous mesh called glassy carbon by solid phase heat treatment (carbonization) processes at temperatures close to 1000 ºC (BENTO, 2004). Glassy carbon has a structure that is more closely related to a non-crystalline material, with high gloss and fracture characteristics such as glass, hence the glassy name, therefore, not having a regular ordered structure, which is an inconvenient factor for several reasons, the main one being the prevention of graphitization (heat treatment at temperatures above 2000º C). Glassy carbon is also often referred to as polymeric carbon, since it derives mainly from the carbonization of polymeric precursors (JENKINS; KAWAMURA, 1976). The carbon materials obtained from thermo rigid resins based on phenolic resins have a specific mass of approximately 1.50 g/cm^3.

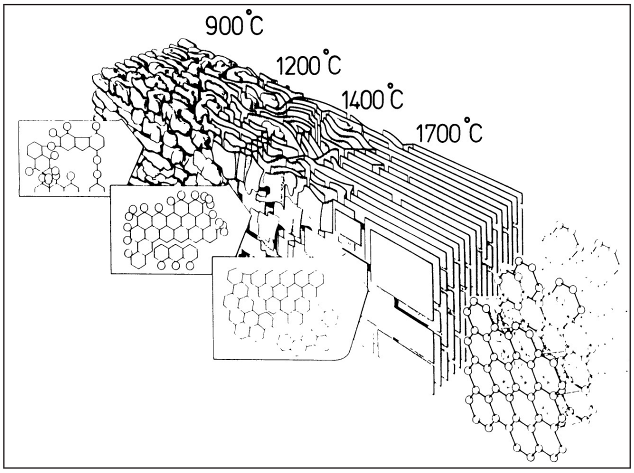
The processes of obtaining carbons in the liquid phase by thermoplastics include the use of tar from petroleum tar or coal tar (PARDINI; GONÇALVES, 2009). The tars are semi-solid viscoelastic materials. Under thermal treatment (temperatures around 550-600 °C), they pass through a liquid phase, with a minimum viscosity, becoming an infusible and thermo rigid material called semicoke. In this case, if the heat treatment is carried out up to 1000° C and at atmospheric pressure (0.1 MPa), the mass loss of the tar, equivalent to 50% of the initial material, occurs, whereas under thermal treatment and at higher pressures at 50 MPa, the yield of the carbon material may be greater than 80% by mass (SOHDA; SHINAGAWA, ISHII, 1999). In general, the process takes place by the thermal conversion of the tar into graphite material, Figure 6, in which the amorphous (tar) material is continuously organized by temperature, by simultaneous flow and alignment of macromolecules, which in turn are arranged and ordered, generating stacked and well-oriented graphical basal planes (GRIFFITHS; MARSH, 1981; RAND, 1993; YOUNG-JAE; HYEOK JONG, 2004), as shown in Figure 7. The carbon materials obtained with graphitized tars have a specific mass greater than 1.9 g/cm^3.

Figure 6 – Thermal conversion of the tar into graphite.



Source: Levy Neto and Pardini (2016, p. 63).

Figure 7 – Illustration of the tar graphitization evolution from the pyrolysis.



Source: Marsch and Rodríguez-Reinoso (2006) and Savage (1993).

In summary, the solid phase heat treatment of thermo rigid resins, such as phenolic resins, generates non-graphitizable carbons, consequently the thermomechanical properties are not satisfactory for most applications. The use of high pressure process for thermo rigid materials does not change the carbon yield. On the other hand, the pyrolysis liquid phase of tars results in highly oriented and graphitized carbons with better thermomechanical properties, having the disadvantage of being carried out at high pressures, considering that the carbon yield of tars is a function of the pressure process (PARDINI; GONÇALVES, 2009; SAVAGE, 1993).

In gas-phase processes, called CVI/Chemical Vapor Infiltration (CVI) or Chemical Vapor Deposition (CVD), hydrocarbon gases such as methane, propylene and others with high carbon content in the molecule, as well as vaporizable liquids, for example cyclohexane, kerosene and others, are subjected to a thermal decomposition process at temperatures ranging from 800 to 1200 ºC, leading to the deposition of carbon in the preform (PARDINI; GONÇALVES, 2009). Therefore, in the CVI method, the gaseous reagents infiltrate the preform, maintained at high temperatures, depositing the mesh material on the fiber structure by means of vapor deposition reactions (CVD). As the infiltration occurs, the CVD deposits continuously grow to form the composite mesh. The CVI process results in the obtainment of graphitizable material, called pyrolytic carbon.

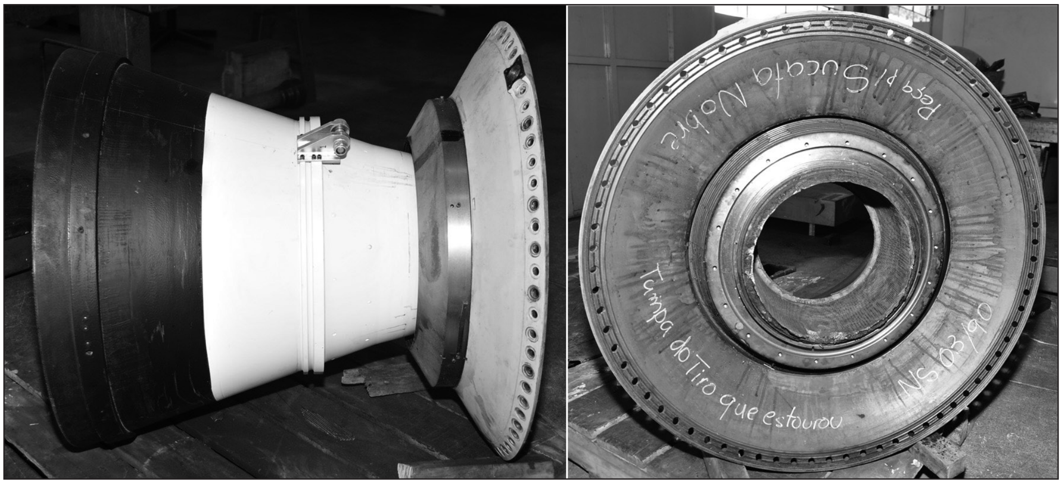
A rocket nozzle throat, manufactured in CRFC composite, used in the IAE engine S43, was used for the analysis in the present work, according to Figure 8.

Stereomicroscopy, optical microscopy and scanning electron microscopy analyzes were performed on samples taken from the multidirectional CRFC composite material in the convergent, throat and divergent regions.

1.1.3 Stereomicroscopy

The juxtaposition of the Greek terms *stereo* relating to two (double), and *scopos*, relative to the view (observer), results in the term stereoscopy, which refers to the visualization of the same focus by two mechanisms of image capture. In general, when in human beings, it is said that the image perceived by the brain results from the combination of two images captured, one in each eye. This pair of pictures is called stereoscopic pair (stereo image pair).

Figure 8 – S43 engine nozzle.



Source: The author.

A stereo is an optical microscope that works with increases from 10X to 90X or up to 180X with the addition of optional extra lenses. It works by using two complete microscopes, inclined one to the other at an angle of 8 to 12 degrees, depending on the manufacturer. Each microscope includes a lens, an eyepiece and a construction system, the latter being either reflective or refractive type. The two lenses and the two eyepieces give the eyes slightly different viewing angles (Figure 9a). In essence, the left and right eyes view the same object, but in a different way. Much like what happens to human eyes, these two separate viewing angles produce a three-dimensional image, which makes it ideal for examining surfaces of solid materials. The lighting is also different compared to other types of microscopes. It uses reflected or episcopic light to illuminate specimens. This means that it uses light, naturally reflected from the object. This is ideal when it comes to thick or opaque samples. The equipment is shown in Figure 9b.

1.2 Optical Microscopy (OM)

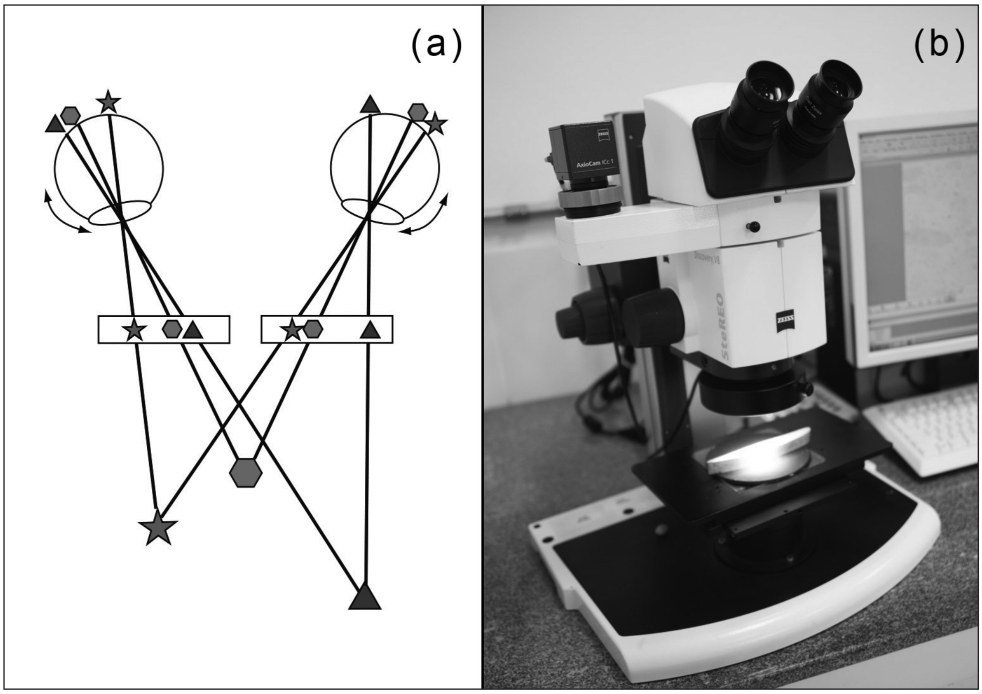
The analysis by optical microscopy allows the evaluation of material sections, allowing the visualization of fibers' arrangement and the existence of defects, such as pores, inclusions and microcracks. Carbon materials are generally observed by reflection with the aid of an optical microscope due to their high absorption coefficients at visible wavelengths. Therefore, most optical studies on CRFC composites use reflection in polished samples (SAVAGE, 1993). The optical microscopy technique consists of characterizing the materials by observing the image generated by the interaction of a collimated light beam with the polished surface of the sample. The signal generated by the interaction between the source and the sample passes through an optical system to obtain an image that is collected, stored and subsequently interpreted.

1.3 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is an analysis technique that expands the sample size for visualization of its structures and topography of solids. Unlike optical microscopy, which uses a light source (photons) for image observation, the SEM uses an electron source increasing the resolution of the images. An important feature of SEM is the three-dimensional appearance of the sample image. It also allows the examination in small increments and with great depth of focus, which is extremely useful, since the electronic image complements the information given by the optical image (DEDAVID; GOMES; MACHADO, 2007).

The principle of SEM is based on the focusing of an electron beam passing through a column under vacuum through a series of electromagnetic lenses (coils) for beam collimation and focusing in a given region of the sample.

Figure 9 – Stereomicroscope. (a) Optical scheme; and (b) Equipment.



Source: Russ (2011).

2 MATERIALS AND METHODS

The **afterburn** evaluation of the S43 engine nozzle insert material was performed by applying microscopic analysis techniques after the engine fire test. The solid propellant used in the S43 engine is the high solids composite type which results in gases at high temperatures. The resin used is hydroxylated polybutadiene, mixed with powdered aluminum and ammonium perchlorate.

Stereomicroscopy, Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) were used for characterization and also for the evaluation of damages caused by surface firing of test specimens.

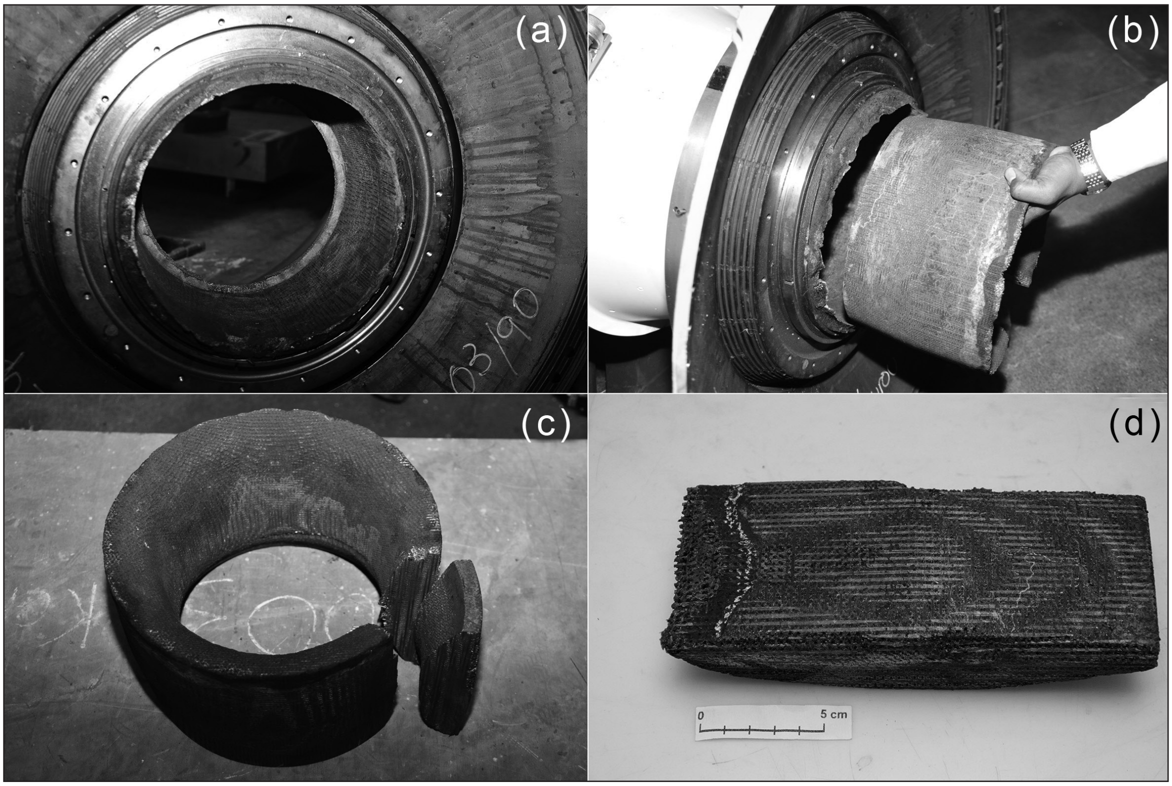
2.1 Materials

Samples of the CRFC composite material were evaluated in the convergent, throat and divergent regions. The process for obtaining the samples followed the steps described in items 2.1.1 and 2.1.2.

2.1.1 Removal of S43 nozzle samples for analysis

Removal of the nozzle insert was performed according to Figures 10a to 10d. It can be seen in Figure 10a that the insert/heat shield is mounted on the engine nozzle S43; in Figure 10b the insert is removed from the frame; in Figure 10c the removal of a section of the insert for analysis; and in Figure 10d the insert sample was removed for analysis.

Figure 10 – Removal of the S43 nozzle insert/thermal protection sample. (a) Thermal protection mounted on the S43 engine nozzle; (b) Thermal protection being extracted from the structure; (c) Removal of a longitudinal section for analysis; (d) Detail of the sample removed for analysis.

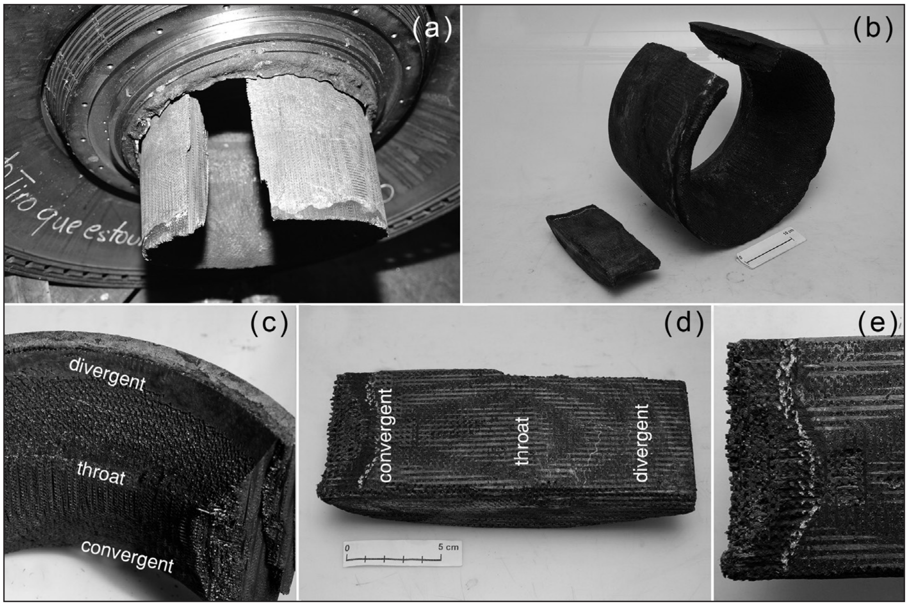


Source: The author.

2.1.2 Subdivision and identification of the SPT sample of the S43 nozzle

In the Figure 11a a sample from the insert/thermal protection mounted on the S43 engine nozzle was removed for analysis. Figure 11b shows an image to the left of the sample and to the right of the insert shown in Figure 11a. For the characterization of the sample shown in Figure 10d and Figure 11b, the three internal regions of the insert (convergent, throat and divergent) were identified, both in the insert, Figure 11c, and in the sample, Figure 11d. Figure 11e shows an enlarged image of the ablative effects of the thermal jet coming from the burning of the propellant in the convergent thermal protection.

Figure 11 – IIdentification of thermal protection regions. (a) Sample for analysis; (b) Internal protection region; (c) Detail of the convergent region; (d) Sample removed from the engine nozzle; (e) Identification of the regions of the insert material.



Source: The author.

2.1.3 Sample cutting process

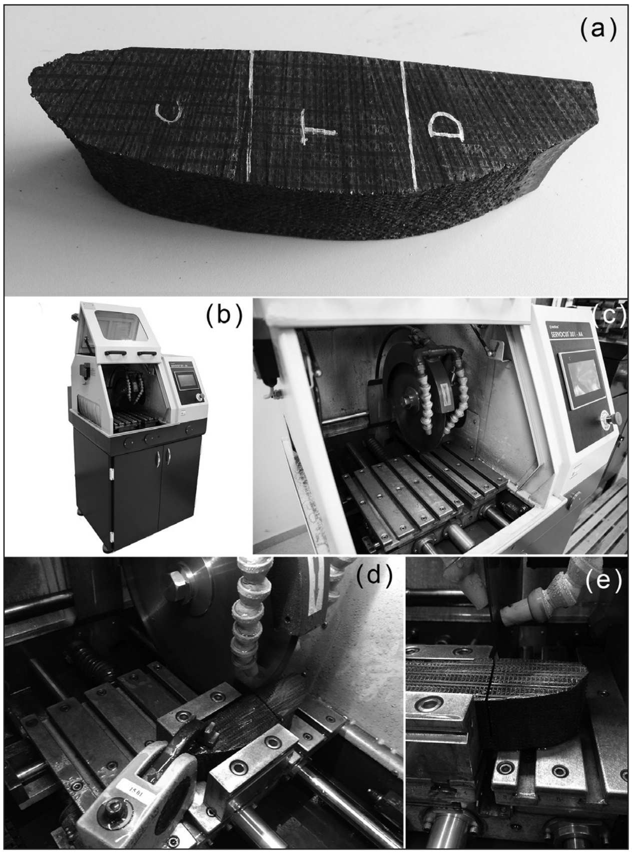
It was convened in the cutting/subdivision process of the thermal protection of the S43 insert sample, Figure 12a, identify it with the letters **C**, **T** and **D**, being designated, respectively, the Convergent, Throat and Divergent. Figures 12b and 12c show the positioning of the sample in the SERVOCUT mod. 301-AA of METKON and, in Figures 12d and 12e, the cutting.

After separating the convergent **C** region of the sample, identified in Figure 12a, the cut was made that allowed to separate the samples of the region of the throat **T** and the divergent **D**, Figure 13a. After the cutting operation, the three samples were oven dried at 70 ºC for 30 min, as shown in Figure 13b.

In order to systematize the analysis, a quadrilateral identification was performed, according to Figure 14a, in perspective, and Figure 14b, in an upper image of the sample, after the drying of the three samples.

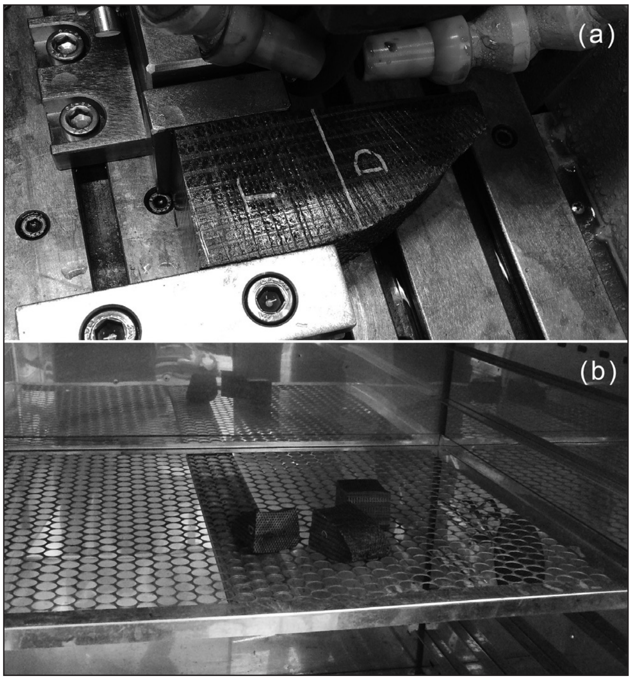
However, the need to perform an additional cut along the thickness of the samples was observed in order to obtain a flat surface with adequate dimensions to assemble in the sample holder of the microscope.

Figure 12 – Sample cut. (a) Identification for cutting; (b) Equipment used; (c) Slot in the specimen fixation and cutting equipment; (d) and (e) Fixation and cutting the **C** convergent of the sample.



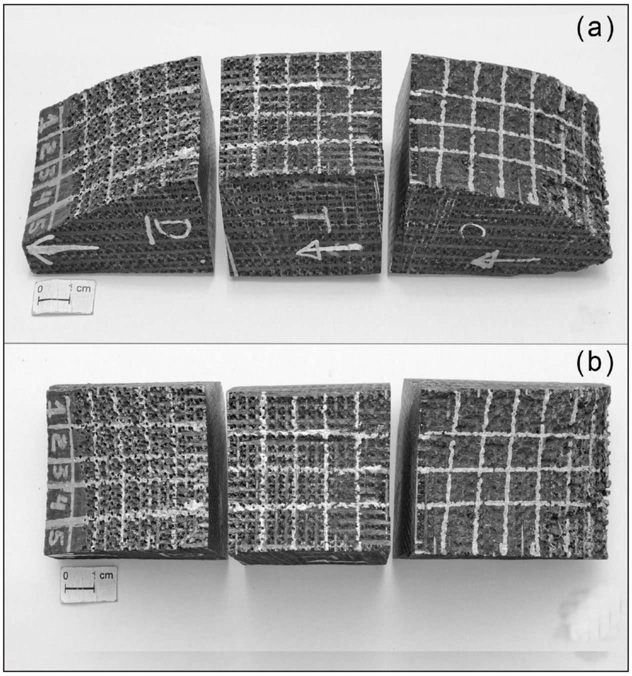
Source: The author.

Figure 13 – Samples. (a) Final cut of the sample; (b) Drying in an oven.



Source: The author.

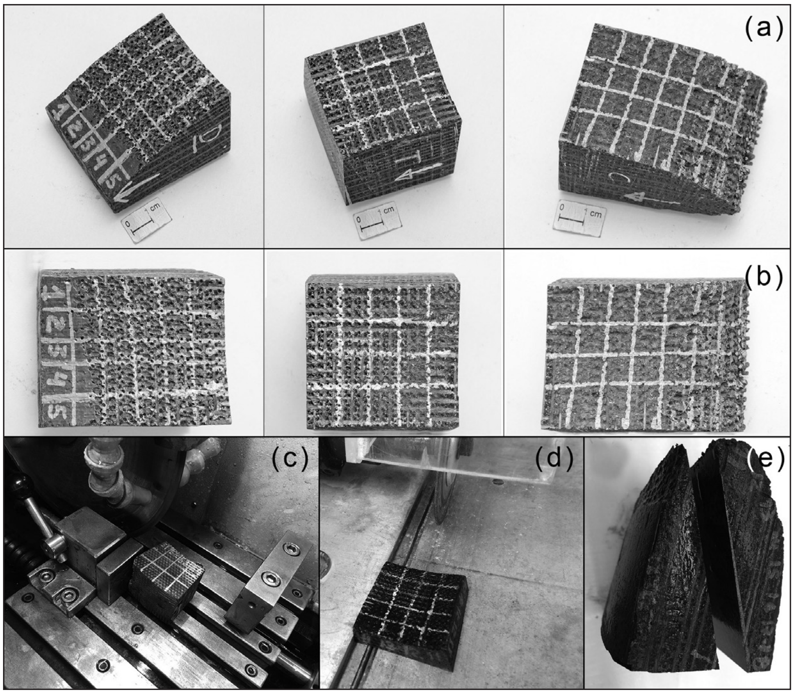
Figure 14 – Demarcation in quadrants of the sample. (a) Perspective image; (b) Top View.



Source: The author.

In Figure 15a there is a perspective image of the divergent **D**, Throat **T** and Convergent **C** samples. In Figure 15b, an upper image of the samples shown in Figure 15a. In Figure 15c, a longitudinal section of the throat region is shown, and in Figure 15d, the section obtained after sectioning the throat sample. In Figure 15e there is the sample of the convergent **C**, which was also submitted to an angular cut to obtain a flat surface.

Figure 15 – Cutting of the sample of the throat **T**, divergent **D** and convergent **C**. (a) Perspective image of the throat; (b) Top View; (c) Cutting of the throat sample in half; (d) Sample obtained from the throat; (e) Divergent and convergent samples; (f) Sample obtained from the convergent.



Source: The author.

3 CONCLUSION

Key concepts of thermal protection materials for launch vehicles were presented. These thermal protections are internal or external to the vehicles and are composed of ablative materials, represented by the polymeric composites or by reirradiant materials, represented by the thermo-structural composites.

A rocket nozzle throat manufactured in CRFC composite, a component of the IAE/DCTA S43 vehicle, was analyzed. This thermal protection, internal to the launch vehicle, can demonstrate ablative and reirradiant characteristics.

The procedures for the extraction of the CRFC composite insert from the S43 engine were systematized. The material analysis strategy was performed by cutting the insert into three sections corresponding to the convergent, divergent and throat regions (critical section).

The analyses were performed by stereoscopy and optical microscopy.

The analyses should indicate the characteristics of erosion and systematize the procedure for analyzing the material behavior, in the face of the operating conditions to be presented in Part II of the work.

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REFERENCES

BENTO, M. S. **Estudo cinético da pirólise de precursores de materiais carbonosos**. 2004. Dissertação (Mestrado) - Instituto Tecnológico de Aeronáutica. São José dos Campos: ITA. 215 p. Disponível em: http://www.bdita.bibl.ita.br/tesesdigitais/lista_resumo.phpnum_tese=000530317. Acesso em: 6 jul. 2018.

DEDAVID, B. A.; GOMES, C. I.; MACHADO, G. **Microscopia eletrônica de varredura**: aplicações e preparação de amostras: materiais poliméricos, metálicos e semicondutores. Porto Alegre: EDIPUCRS, 2007.

FITZER, E.; MANOCHA, L. M. **Carbon reinforcements and carbon/carbon composites**. Berlin: Springer-Verlag, 1988. 343 p.

GONÇALVES, A. **Caracterização de materiais termoestruturais a base de compósitos de carbono reforçados com fibras de carbono (CRFC) e carbonos modificados com carbeto de silício (SiC)**. 2008. Tese (Doutorado) – Curso de Pós-Graduação em Engenharia Mecânica e Aeronáutica. Instituto Tecnológico de Aeronáutica. São José dos Campos: ITA. 226 p. Disponível em: http://www.bdita.bibl.ita.br/tesesdigitais/lista_resumo.php? num_tese=000549057. Acesso em: 6 jul. 2018.

GRIFFITHS; J.A.; MARSH, H. **Proceedings of 15th Biennial Conf. on Carbon**. University of Pennsylvania, Philadelphia, USA, 1981. p. 22-26.

JENKINS, G. M.; KAWAMURA, K. **Polymeric carbon**: carbon fibre, glass and char. Cambridge: Cambridge University Press, 1976.

LAUB, B.; VENKATAPATHY, E. Thermal protection system technology and facility needs for demanding future planetary missions. In: INTERNATIONAL WORKSHOP PLANETARY PROBE ATMOSPHERIC ENTRY AND DESCENT TRAJECTORY ANALYSIS AND SCIENCE. **Proceedings**... Noordwijk: ESA Publications Division, 2003.

LEVY NETO; F.; PARDINI, L.C. **Compósitos estruturais**: ciência e tecnologia. 2.ed. São Paulo: Blucher, 2016. 418 p.

LIUYANG, D.; XING Z.; YIGUANG, W. Comparative ablation behaviors of C/SiC-HfC composites prepared by reactive melt infiltration and precursor infiltration and pyrolysis routes. **Ceramics International**, 43, p. 16114–16120, 2017.

MARSH, H.; RODRÍGUEZ-REINOSO, F. **Activated carbon**. Amsterdam: Elsevier, 2006.

PALMERIO, A. F. **Introdução à tecnologia de foguetes**. São José dos Campos, SP: SindCT, 2017.

PARDINI, L.C.; GONÇALVES, A. Processamento de compósitos termoestruturais de carbono reforçado com fibras de carbono. **Journal of Aerospace Technology and Management**, São Paulo, v. 1, n. 2, p. 231-241, jul. - dez. 2009.

PULCI, G., et al. Carbon–phenolic ablative materials for re-entry space vehicles: manufacturing and properties, **Composites A** 41, p. 1483–1490, 2010.

RAND, B. **Matrix precursors for carbon-carbon composites**, Essentials Carbon-Carbon Composites, Chap.3, Royal Soc. of Chemistry, London, UK, 1993. p. 67–102.

RUSS, J. C. **The Image Processing Handbook**. Sixth Ed, CRC Press, 2011. p.70.

SAVAGE, G. **Carbon/carbon composites**. London: Chapman & Hall, 1993. 389 p.

SILVA, H. P.; PARDINI, L.C.; BITTENCOURT, E. Shear properties of carbon fiber/phenolic resin composites heat treated at high temperatures. **Journal of Aerospace Technology and Management** 8, n.3, p. 363-372, 2016.

SILVA, W. G. **Qualificação de materiais utilizados em sistemas de proteção térmica para veículos espaciais**. Tese (Mestrado em Física dos Plasmas) - Instituto Tecnológico de Aeronáutica – ITA, 2009. 112 p. Disponível em: http://www.bdita.bibl.ita.br/tesesdigitais/lista_resumo.php?num_tese=000555112. Acesso em: 6 jul. 2018.

SOHDA, Y.; SHINAGAWA, M.; ISHII, M. Effect of carbonization pressure on carbon yield in a unit volume. **Composites Part A**: Applied Science and Manufacturing, v. 30, n. 4, p. 503-506, Apr. 1999.

SUTTON, G. P. **Rocket propulsion elements**. 8. Ed. New York: Wiley, 1992.

THIYAGARAJAN, N. **Processing and characterization of reaction formed SiC - based ceramic matrix composites**. Madras: Anna University, 1996.

WITTMANN; K.; Fundamentals. In: WILFRIED L, WITTMANN K, HALLMANN W. (Ed.). **Handbook of Space Technology**. United Kingdom: John Wiley and Sons, 2009. p. 33-113.

YONG-JIE, W. et al. Ablation behavior of a TaC coating on SiC coated C/C composites at different temperatures, **Ceramics International**, 39, p. 359–365, 2013.

YOUNG-JAE L.; HYEOK JONG J. Investigation on ablation behavior of CFRC composites prepared at different pressure. **Composites: Part A** 35,1285–1290, 2004.