DOKUZ EYLÜL UNIVERSITY

GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

DYNAMIC FRICTION BEHAVIOR OF HIGH STRENGTH COMPOSITE MATERIALS

by

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M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled **"DYNAMIC FRICTION BEHAVIOR OF HIGH STRENGTH COMPOSITE MATERIALS"** completed by **EGE KIZILAY** under supervision of **PROF.DR. ÇINAR EMİNE YENİ** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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DYNAMIC FRICTION BEHAVIOR OF HIGH STRENGTH COMPOSITE MATERIALS

ABSTRACT

Composite materials with low weight and high strength are increasingly used in many land, sea, air and space vehicles, especially in the outer surface coatings of such vehicles. When aircraft and spacecraft are considered, composite materials used on exterior surfaces are exposed to dynamic friction due to the fact that very high speeds are reached in a short time. Investigating the friction behavior of composite materials with different properties is important in terms of giving the desired performance during the use of the vehicle.

In this thesis study, using a ceramic fiber plate with an insulated layer was used to represent the infrastructure of a composite material with similar properties to the composite materials used in aircraft (Reinforced Carbon Carbon (RCC), High Temperature Reusable Surface Insulation (HRSI) and Low Temperature Reusable Surface Insulation (LRSI). The most appropriate insulation material was determined by comparing the dynamic friction conditions and mechanical strength of the surface insulation materials produced in sintering furnaces with a layer of ethyl silicate resin with 1750°C and silicon carbide and boron carbide-based 'Ultra High Temperature Ceramics' on the ceramic fiber plate.

Keywords: Boron carbide, silicon carbide, high temperature ceramics, dynamic friction coefficient

YÜKSEK MUKAVEMETLİ KOMPOZİT MALZEMELERİN DİNAMİK SÜRTÜNME DAVRANIŞLARI

ÖZ

Düşük ağırlık ve yüksek mukavemete sahip kompozit malzemeler, başta araçların dış yüzey kaplamaları olmak üzere birçok kara, deniz ve hava aracında giderek daha fazla kullanılmaktadır. Uçaklar düşünüldüğünde dış yüzeylerde kullanılan kompozit malzemeler çok yüksek hızlara kısa sürede ulaşılmasından dolayı dinamik sürtünmeye maruz kalmaktadır. Farklı özelliklere sahip kompozit malzemelerin sürtünme davranışlarının araştırılması, aracın kullanımı esnasında istenilen performansın verilmesi açısından önemlidir.

Çalışmada, uçaklarda kullanılan kompozit malzemelere (Reinforced Carbon Carbon (RCC), Yüksek Sıcaklıkta Yeniden Kullanılabilir Yüzey İzolasyonu (HRSI) ve Düşük Sıcaklıkta Yeniden Kullanılabilir Yüzey İzolasyonu) benzer özelliklere sahip bir kompozit malzemenin seramik fiber plaka altyapısı kullanılarak (LRSI), sinterleme fırınlarında üretilen yüzey yalıtım malzemelerinin 1750 santigrat derece dayanımlı etil silikat reçine tabakası ile silisyum karbür ve bor karbür esaslı 'Ultra Yüksek Sıcaklık Seramikleri' ile dinamik sürtünme koşulları ve mekanik dayanımları karşılaştırılarak en uygun sonuç analizi yapılmıştır.

Anahtar Kelimeler: Bor karbür, silisyum karbür, yüksek ısı seramikleri, dinamik sürtünme katsayısı

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CHAPTER ONE

INTRODUCTION

1.1 Source of Inspiration

When we examine the vehicles that carry us from our location to another place, we are generally interested in how their interior parts or appearances are, and often we want to have more information on these issues. If we touch on this subject with a different perspective, you will understand that the exterior design of the vehicle you are actually using has been thought and designed in detail so that you can have the most comfortable journey. Because in sea, land, air and space journeys, the comfort of the vehicle is not evident on a straight road without obstacles, but at a point full of bumps and difficulties.

If we think about these difficulties and obstacles in aircraft and spacecraft, you may not be able to think of anything particular that these vehicles may come across. After all, we see these vehicles floating in the air with no obstacles in front of them, when we look from below. But here, too, many factors such as temperature differences due to altitude, friction factor due to vehicle speed, and aircraft weight etc. affect the flight comfort.

The space race which emerged with the end of the Second World War that started in the second half of the 1940's helped the two world giants, America and Russia, to pioneer many technological advances around the world. Examples of these are the first satellite launched from the Earth into space, the first launched of a living animal, the launch of a human to space, and finally the first manned flight to the moon (Wikipedia- Apollo 11). If we look at the phenomenon considered to be the first priority in these flights and examine the sequence of events, the priority has always been the human health and safety. One of the factors affecting human safety during a flight is the resistance and endurance of the vehicle in the face of difficult situations. The situation that a spacecraft encounters and that can affect human life the most is the temperature difference and friction factor, as mentioned above. If we examine these two elements under a common title, we can present the ultra-high friction force that occurs between the high-speed spacecraft and the atmosphere, which a spacecraft experiences during its exit and entry from and into the atmosphere, as a common title. In Figure 1.1, an image showing the moment of entry of a spacecraft into the earth's atmosphere is given.

Figure 1.1 Representative illustration of a space shuttle's entry into the atmosphere (Jonas Dino-2008)

1.2 Review

1.2.1 Objective

If we examine the outer designs of space vehicles; it is necessary to analyze the heat tiles used in space shuttles. If heat tiles are to be examined basically, they are referred to as composite materials. Composite materials are increasingly used in air and space vehicles as well as land and sea vehicles due to their superior properties such as being easy to form, light weight, possessing high strength values and high corrosion resistance. The use of composite materials has advantages in many stages, from new product design to the development of an existing product, from ease of use to maintenance and repair. In addition, the use of fewer parts compared to metallic materials and the low labor costs make composite materials preferable in sea, land and air vehicles.

In the aerospace industry, which ranks among the first among the composite usage areas, composite materials are used in aircraft and helicopter interior design, structural material production and exterior surface coatings based on their superior mechanical properties as well as their lightness (NASA.gov/ Lightweight Spacecraft Materials and Structures- 2014).

Considering air and spacecraft composite materials used on exterior surfaces are exposed to dynamic friction due to the fact that very high speeds are reached in a short time. In addition, spacecraft flying at high altitudes are exposed to severe temperature changes. For example, there is a large temperature difference of several hundred degrees Celcius between the sun-facing and non-solar side of a spacecraft. Investigating the friction behavior of composite materials with different properties is important in terms of giving the desired performance during the use of the vehicle (blog.keronite.com/the-lightweight-future-of-space-design-and-material-selection- 2023).

Reducing mass is one of the most important criteria in aircraft design. Reducing the structural mass will contribute to the reduction of the vehicle's manufacturing costs and fuel required for flight, as well as extending the life of the vehicle. The weight of the aircraft is the common factor that links all aspects of aircraft design such as aerodynamics, structure, and propulsion, all together. An aircraft's weight is derived from various factors such as empty weight, payload, useful load, etc. The various weights are used to then calculate the center of mass of the entire aircraft. The center of mass must fit within the established limits set by the manufacturer. In addition to traditional materials such as aluminum used in space applications, Mg-Li alloys have come to the fore in recent years, but the precision machining of the surfaces of these two materials is extremely difficult and they have the possibility of reacting with components in space. Composite materials come into play as an alternative at this point.

It is desirable that the outer surface materials used in spacecraft have features and standards far superior to those used in land and sea vehicles. Such vehicles are extremely difficult to access and maintain, and the conditions to which they are subjected are much more demanding. The importance of the insulation materials coated on the outer surfaces of the composite materials, which are used as outer surface coatings especially in space crafts, are also revealed here. It is these materials coated on the composite material surface that increase the actual frictional resistance and improve the ability to withstand temperature.

In this thesis, it is aimed to examine the dynamic friction behavior and wear of these materials by producing composite materials similar to those used in the outer coatings of space vehicles, and to determine the most suitable one among them by comparing the friction and wear behavior of composite materials with different components. Composite materials envisaged to be used in this study will be materials with properties similar to Reinforced Carbon-Carbon, reusable surface insulation at high temperatures and reusable surface insulation materials at low temperatures. A visual of High-temperature reusable surface insulation (HRSI) is given in Figure 1.2. In Figures 1.3 and 1.4, schematic and various visuals of heat tiles are included.

Figure 1.2 High-temperature reusable surface insulation (HRSI) (Magnus Manske-2013)

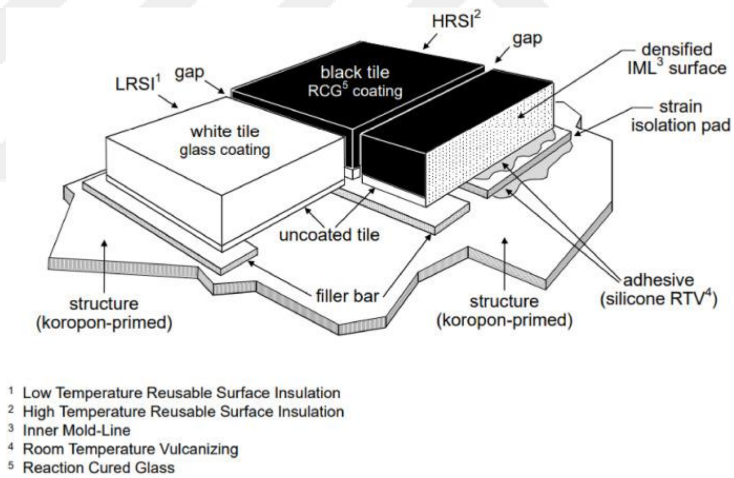

Figure 1.3 Schematic of the common structure of heat tiles (Obinna Uyanna & Hamidreza Najafi- 2020)

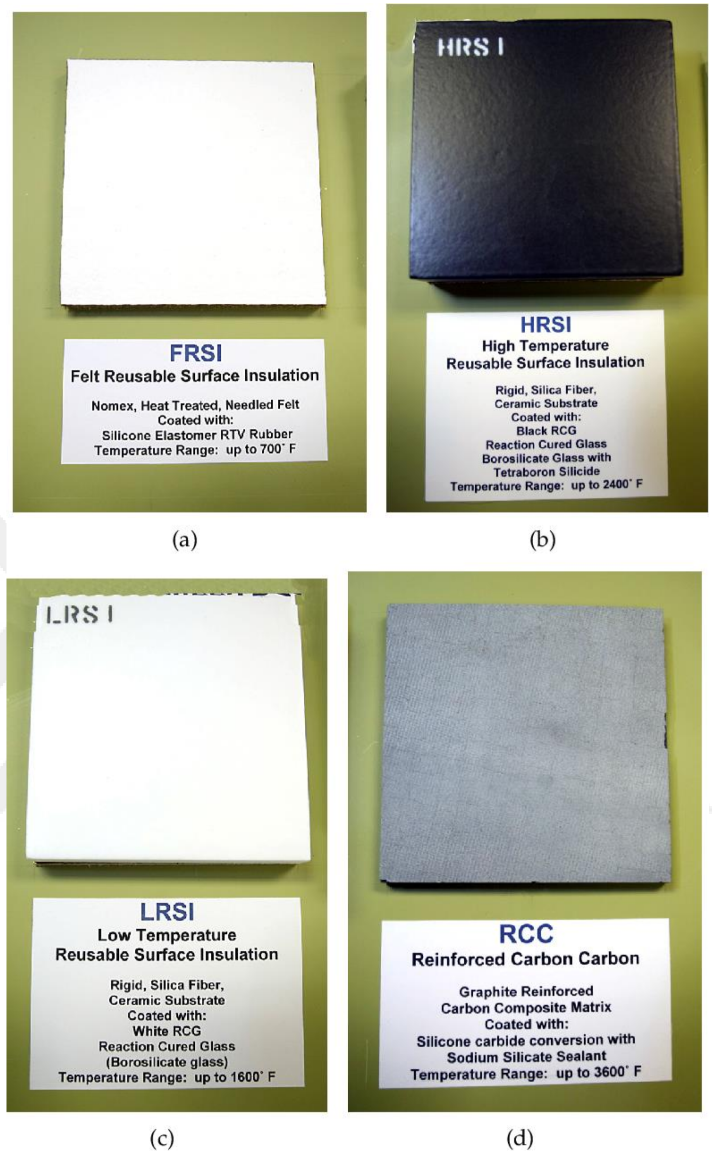

Figure 1.4 Collective image of heat tiles used in space shuttle systems (W. Ng- 2005)

1.2.2 Target

Spacecraft must operate in a high vacuum environment for long periods of time. One problem designers will encounter in this context is the significant increase in friction. Frictional forces that must be taken into account both during the flight of the vehicle, when its outer surface is in contact with space at high speed, and during the vehicle's mission with the substances in the outer space (these substances called pollutants are natural oxides in the atmosphere, organic substances, etc.). In addition, maneuvers using gravity during the landing of spacecraft on a space object are also one of the frequently used landing methods. However, in this case, the spacecraft is exposed to significant frictional forces created by the atmosphere layers when landing on the earth (or another space object).

When vehicles rising from the earth and entering the atmosphere are exposed to atmospheric friction forces, these forces apply mechanical stress on the objects, and also aerodynamic heating occurs as a result of the compression of the air in front of the object and again due to atmospheric friction. These forces can cause loss of mass or even fragmentation of small objects. Objects with low compressive strength can burst with explosion. For this reason, the outer surface coatings of vehicles planned to operate in a vacuum environment should have a structure that minimizes friction forces.

In addition, since the vehicles operating in the space environment are exposed to great temperature changes, the materials used on the outer surfaces of the spacecraft must be heat resistant.

The aim of this study is to examine the dynamic friction and wear behavior of these materials by using different materials coated on composite surfaces similar to those used on the outer surfaces of space crafts and to choose the most suitable one among these materials. For this purpose, it is planned to coat ceramic powders with superior wear resistance on high temperature resistant ceramic fiber board material. The mechanical properties of these materials, which will be coated with the spraying method on ceramic fiber board in specified thicknesses, will be determined first and then the friction and wear will be measured at different speeds by using the tribometer device with the ball-on-disk method. It will be determined which material or materials show better friction behavior throughout the process and process improvement will be made if necessary.

The ultimate goal of the proposed research is to contribute to the development of outer surface coating materials that will have the least friction resistance in a vacuum environment, within the scope of space studies, which is among the priority targets of our country. A representative image of the tribometer is given in Figure 1.5.

Figure 1.5 Tribometer (Margaret Stack- n.d.)

1.2.3 Scope

The importance of composite materials, which are used in spacecraft and prevent the access of the heat released as a result of dynamic friction between the atmosphere and the spacecraft to the inside of the vessel and can absorb the strength of the damage that may occur (impact, wear due to dynamic friction, etc.), has been revealed as a result of several researches. In order to prevent the loss of life and property in the designed spacecraft, the thermal insulation design should be put forward as a result of very successful engineering studies.

As can be understood, the composite materials used in spacecraft can have a direct impact on human safety. Another importance is due to economic reasons. Here, the lightness and strength of composite materials come to the fore. Considering that each gram of excess mass will cause thousands of dollars in losses, especially in aircraft, composite materials are considered as a good way out for this.

In terms of strength, the results of the literature research vary according to the coating material to be used and its ratio. Resistance to friction as a result of dynamic friction between the atmosphere and high-speed spacecraft, resistance to corrosion for repeated and long-term use, high impact resistance against the possibility of collision with space stones and dust, and as it can be summarized, the coating material to be designed must be high strength despite harsh space and atmospheric conditions required. On the other hand, as a result of the investigations made within the scope of the thesis, the following results were obtained regarding the thermal ceramics used in the shuttle systems; a production phase was followed as a high-strength ceramic coating on the heat-proof (small amount-not adversely affecting human health) plate and subjecting it to the sintering process.

Within the scope of the thesis, it has been been aimed to produce a product that can reflect these features in the basic sense under academic production conditions and to be an area open to development and improvement in the future. The mechanical tests to be carried out on the product which will be revealed will shed light on our observation of the results in terms of suitability and susceptibility for the purpose of production. The importance of the research for our country highlights the research question part of the project. As a result of the researches, it has been understood that there are studies on coating materials, but it is not directly related to the subject of thermal ceramics used in spacecraft.

1.3 Literature Research

1.3.1 Uygunoğlu, Brostow and Gunes (2015) Wear and Friction of Composites of an Epoxy with Boron Containing Wastes

The friction and wear behavior of the composite material obtained by using dibor trioxide, one of the varous boron added compounds obtained from Kırka Boron Factories, was evaluated. The results obtained from the use of different boron minerals at different rates will be discussed in the following sections. In this article, the important part for our thesis is the section in which the dynamic friction behavior is examined tribologically and the result obtained that the boron mineral additive has a positive effect on friction.

In the following part of the introduction, the positive aspects of the effects of these wastes on wear and friction, which are used as a satisfactory filler for epoxy resin, are mentioned. Polymer materials have been affirmed in many ways. The important part for us here is the part mentioned above.

Table 1.1 Chemical ratio of boron wastes used

Oxide	B_2O_3	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	Fe_2O_3	Loss of Ignition (LOI)
Content, %	12.09	15.5	1.38	17.7	13.79	0.5	3.34	0.22	34.4

In the light of the data used in Table 1.1, the wear and friction behaviors of the nanoparticles to be used in the epoxy resin mixture were prepared and used within the scope of our thesis, especially in relation to Diboron Trioxide/Boron Oxide and Silicon-Silicon Dioxide, which are used more proportionally than other materials. It can be concluded that positive opinion has been obtained.

Table 1.2 Mixture and hardness states of the composite

Mixture code	Epoxy resin*, kg/m³	Boron waste, kg/m³	Hardness (H_D)
B0	100	-	32
B10	90	10	38
B20	80	20	47
B30	70	30	59
B40	60	40	74
B50	50	50	98

*Epoxy resin was used with the hardener in the 2:1 ratio.

As can be seen in Table 2.1.2, that the amount of boron waste material that is regularly increased as content in the epoxy resin used increases the hardness.

Table 1.3 Dynamic friction values under different conditions

Mixture code	5.0 N	10.0 N	15.0 N
B0	0.37	0.39	0.40
B10	0.28	0.33	0.36
B20	0.26	0.30	0.32
B30	0.21	0.27	0.30
B40	0.19	0.22	0.24
B50	0.16	0.19	0.21

As can be seen in Table 1.3, the dynamic friction value decreases as the amount of boron waste material increases.

Differences with our study:

1- The friction and wear behavior of the material was investigated by using only a mixture of one material in different proportions and not using it at all. We, on the other hand, will use different active ingredients in our study and will go to a comparison and optimization path.

2- In our thesis, a composite study will be carried out on ceramic fibers with high heat resistance and low heat permeability with the coating method –coating spray-method.

1.3.2 Luangtriratana, Kandola and Myler (2015) Ceramic Particulate Thermal Barrier Surface Coatings for Glass Fibre-Reinforced Epoxy Composites

In the aforementioned article, an experimental optimization-based research process for a thermal barrier was carried out with micro-sized ceramic particles containing Nanoclay, Smoky Nanosilica, Glass Flake, Aluminum Titanate and Zirconium. A modified liquid phenol resin named DUREZ 33156 was used as the binding phase.

If we examine the issues that are important for our study, first of all, the use of nanoparticles (silicon carbide, boron carbide, zirconium, aluminum oxide, which we will use in powder form with binder-paint in our project) as a thermal barrier, stands out. In the conclusion part, it was said that the best result was obtained in glass flake material, but it is understood that other intermediate products also gave results suitable for their intended use.

Our second important result is the fact that SpaceX employee Martin Wilson talks about an alcohol-water-mixed adhesive product about the coating fluid in the light of the information he gave in his short video document called Thermal Protection System. Phenol resin, which is also used in the examined article, gives a product idea suitable for these features. Its content consists of 58-78% polymer formaldehyde, 20-29% ethyl alcohol-ethanol and 3% water. The method we will use in our study, on the other hand, will be fabrication, a heat-resistant paint-resin and optimization work with a resin that we will prepare in the light of the above ratios.

Differences with our study;

1- In our study, our working temperature, product resistance temperature is planned to be around 1200°C maximum. In the literature study, the operating temperature is in the range of 180-250 degrees and the service purpose is independent of space studies.
2- The active ingredients to be used in our study to be developed have been mentioned above. However, as a result of the unregistered literature research, it is thought that we can concentrate on zirconium material. Since it is an expensive product, its interaction with other main products can be optimized with its mixed state and its non-mixed state.
3- The product we will use as a coating material named as ceramic fiber-board in the market and it is a material with high temperature resistance. In the reviewed article, GRE composite was used. Considering the thermal conductivity and absorption force of the material we will use in our study, its function for space heat protection shields becomes even more prominent.

1.3.3 Lv, Zheng, Wang (2015) Friction and Wear Behaviors of Carbon and Aramid Fibers Reinforced Polyimide Composites in Simulated Space Environment

The subject of the third article review is the suitability and resistance of reinforced carbon-structured polyimide-composites to the beam-radiation and start-stop friction conditions exposed in space conditions. At the same time, the use and testing of aramid fibers together with carbon fibers and polyimides and their testing is also included in the experiment and conclusion part of the article. The samples used; referred to as CF/PI (Carbon Fiber/ PolyImid) and AF/PI (Aramid Fiber/PolyImide). As a result of the use of the mentioned samples under the harsh conditions of space (AO/UV (Atomic Oxygen/Ultraviolet), irradiations-radiation, radiation- and start-stop friction situations that affect tribological performance) by examining their strength, the sample with which the most efficient result can be obtained has been determined.

One of the important issues is '…The fibers were mixed with PI powder in ethanol with mechanical stirring and ultrasonic bath for 2 h to obtain the uniform mixtures, which were then filtered and dried. The part designated as …'. The ethanol and powder-powder mixture, which was also included in our previous article review, is here again. As it can be understood, the necessity of using ethyl alcohol-ethanol to turn powder-nanoparticle-powder components into a coating/composite component has been able to find support theoretically.

Another point to be noted in the article reviewed is the "Ball-on-Disc"∗5 mechanism. This mechanism, which was used in the examination of friction and wear behavior in the "Friction and Wear Test" part of the research, was also used in the article examined in the first row. The conclusion to be drawn from here; The Ball-on-Disc mechanism is registered as a mechanism that can find theoretical support for the investigation of dynamic friction behaviors within the scope of our project. In Figure 1.6, the images of the samples under the optical microscope are given.

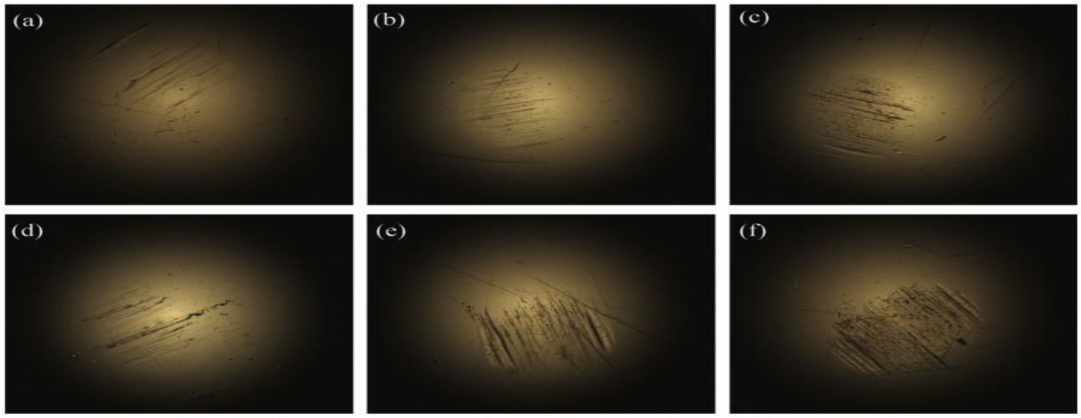

Fig. 10. Optical micrographs of steel ball surfaces: (a) PI, (b) AF/PI, (c) CF/PI, (d) irradiated PI, (e) irradiated AF/PI, and (f) irradiated CF/PI.

Figure 1.6 Examination of samples under an optical microscope (Lv, Zheng and Wang - 2015)

As can be seen in Figure 1.6, the radiation environment has a significant effect on wear. Considering the other tests, it was seen that the most suitable material for use was CF/PI.

Differences with our study;

1- There will be no use of aramid fiber and polyimide materials in our project. Materials such as aramid fiber are generally used in astronaut suits and do not comply with the low density requirement we desire.

2- Tested composite materials are materials in sheet-thin layer. Preparation conditions and products are independent of our study.

3- The tests applied for the conditions of adaptation to the space environment are related to our study, but the material and product purpose used are independent of the project. Note: The tests carried out are in the direction of shedding light on our study. Especially the radiation tests that can be done in the later stages are suitable for our study.

There are several other related studies on friction and wear of composites used in aircraft and spacecraft. Xiao et.al. (2014), investigated the tribological properties of composites containing copper matrix (Cu-MMC) considering the conditions for spacecraft rendezvous and docking operations. They carried out dry friction tests at various temperature (between -100 °C and +100 °C) and pressure values (ambient to as low as 1 10^{-4} Pa) using a multi-disc brake, which is mounted inside a specifically designed simulation system. The test rig was equipped with a temperature control system and pressure regulator. They have calculated the mean friction coefficients (μ_{mean}) for several different conditions like breaking, clutching and docking. They have observed that during breaking the metal matrix composite showed stable friction characteristics at ambient air, while significant fluctuations and increased wear rates were observed in vacuum environment. During clutching operations, there was little sensitivity in tribological performances in both air and vacuum; however, frictional behavior became unstable with the decrease in temperature in vacuum environment. They have concluded that Cu-MMC can reliably perform braking and clutching operations during maneuvering and connection/disconnection in spacecraft.

In a study on the tribological behavior of carbon fiber reinforces ceramic composites (C/C-SiC) used as high frictional materials, Duan et.al. (2021), have examined several factors which affect their friction and wear properties. The factors considered include the content of silicon carbide and carbon matrix, carbon fiber preform architecture and matrix modification by using alloy additives. The influence of various test conditions for C/C–SiC composites were also reviewed. C/C–SiC composites possess good mechanical properties of carbon fiber and outstanding tribological properties and wear resistance of SiC. Owing to their superior tribological behavior, they are used in the fields of aviation, especially in braking systems. C/C–SiC composites possess more stable and higher coefficient of frictions and lower wear rates. They also acquire lower sensitivity to humid and oxidizing environments thanks to the existence of silicon carbide matrix and their relatively reduced carbon content. SiC is a hard phase in ceramic composites, therefore it may cause an increase in the friction coefficient by strengthening the abrasive action during braking operation. Since roughness increases the wear debris may fill in the gaps and get trapped. Also, the formation of a hard friction film leads to a good contact between the friction surface and the mating surface through the film. This causes an increase in the effective contact area. Additionally, relatively a high amount of SiC content and low carbon matrix content could reduce the oxidation corrosion wear of the composite at high temperatures, approximately above 450 °C due to the reduction of carbon oxidation. C/C–SiC composites are insensitive to humid environment due to the weak hydrophilicity of SiC [38]. Therefore slicon carbide content is an important factor considering the friction behavior of such coomposites. It is concluded that, it is necessary to strike a balance between friction and wear performance and mechanical properties of C/C–SiC composites in practical applications.

Taneja (2021), studied the sintering of boron carbide with in-situ silicon carbide formation reaction. Boron carbide is one of the hardest ceramic materials with outstanding hardness. Reaction bonded silicon carbide boron carbide is processed by infiltrating with molten silicon partially sintered mixture of boron carbide particles to obtain a dense composite. Using a binder, the molten silicon infiltrated into the boron carbide pellets and a reaction allowed the formation of hard, solid composites. XRD analyses revealed stable phase formation that depended on temperature. The SEM analysis decreasing porosity was observed with increasing temperature. In a similar study, Li et.al. (2021), a one step sintering of silicon carbide is carried out with the aid of boron carbide. By introducing boron carbide particles into the starting silicon carbide materials, a uniform and high strength substrate was consolidated. When the boron carbide content was increased, the pore size of the substrate decreased, while the flexural strength increased.

Zhang et.al. (2021) present an ultra-high speed ball-on-disc tribometer in this manuscript. In order to attain the ultra-high sliding speed, the y propose a combined solution of on-line precision cutting and in-situ dynamic properties. The ultrahigh-speed ball-on-disc tribometer with a maximum sliding speed of 50 m/s (compared to standard tribometers where the sliding speed is usually lower than 5 m/s) and 120 mm diameter has been developed. This type of tribometer finds application in high speed frictional contacts such as skis and braking of high speed trains. The test apparatus was tried three different kinds of representative engineering materials having different hardnesses and brittleness, namely, high conductivity oxygen-free copper (OFHC), stainless steel and monocrystalline silicon against alumina ball. The friction stability during ultrahigh-speed fricton tests was evaluated to verify the performance of the proposed tribometer.

In an interesting research, series of three dimensional hypervelocity impact simulations have been carried out by Fahrenthold (2006), in order to study the effects of orbital debris impact on the wing leading edge of the Space Shuttle. The authors have developed a new orthotropic elastic-plastic constitutive model for reinforced carbon-carbon (RCC) used in the wing and they have validated their model hypervelocity impact simulation experiments. The authors have applied their model to study RCC perforation at velocities up to 13 km/h. The simulation assumes a fixed projectile mass, but the impact obliquity, the projectile shape and orientation vary in order to study impact conditions of an orbit debris. They have investigated under various circumstances the perforation diameter and the spalled region diameter under impact conditions caused by debris.

CHAPTER TWO

HIGH STRENGTH COMPOSITE MATERIALS

2.1 Selection of Study Materials

While designing the exterior of a spacecraft, as we mentioned earlier, the temperature resistance, thermal conductivity, weight and strength of the material are important. Within the scope of the study we will produce, care should be taken to select a material group that we can transfer these properties to.

2.1.1 Ceramic Fiber Plate

First of all, we should focus on the ceramic fiber plate that we have chosen as the main material which will serve as the base material since this chosen material actually attracts attention with its multitude of usage areas and low density. If the usage areas are to be examined; it is used in high temperature furnaces, boilers, walls, ceilings and doors of industrial furnaces, behind bricks and monolithic refractories, in the transmission of molten aluminum and other non-ferrous metals, against direct flame and heat, against abrasive and air circulation in the furnace atmosphere.

Ceramic fiber plate that we will use as the main material is known for its properties such as; minimum thermal conductivity, resistance to thermal shocks, lightness, minimum shrinkage at high temperatures, resistance to air circulation and chemical gases, physical properties not deteriorating at high temperatures, minimum heat storage, low absorption against molten metals, easy cuttability, sound insulation, not containing asbestos etc. (Seramik Elyaf Plakalar (Board) (n.d.). Retrieved May 18, 2023, from Endüstriyel Malzeme Website)

Features;

- Low thermal conductivity
- Low heat storage
- Lightweight
- Excellent thermal shock resistance
- Resistant to abrasion
- Resistant to most chemicals
- Easy to cut and shape
- Low sound transmission
- Resistant to penetration by molten aluminum and other non-ferrous metals
- Asbestos-free

Usage Areas;

- In the wall, ceiling and doors of industrial furnaces
- In boilers and boilers
- Behind bricks and monolithic refractories
- It is used in the transmission of molten aluminum and other non-ferrous metals, against direct flame and heat, against abrasive and air circulation in the furnace atmosphere. (Ravatherm/ Seramik Yünü, Ceramic Fiber- 2021)

The representative image of the ceramic fiber plate is given in Figure 2.1.

Figure 2.1 Ceramic fiber plate (KGD Website- n.d.)

2.1.2 Silicon Carbide

The second material we will use is Silicon Carbide (SiC), which is the coating material. Silicon carbide is a compound formed as a result of the reduction of silicon dioxide with coke in an electric furnace, when more carbon is used than necessary during the production of pure silicon. Silicon carbide is one of the most widely used structural ceramics. This material is known as high temperature resistant ceramic. The properties of the powder used are that it is 99.9% pure and its grain size is F600 (3 - 19 µm). (Silicon carbide (n.d.). Retrieved May 19, 2023, from Wikipedia)

Silicon Carbide (SiC) and Properties;

- Low density (3.07-3.15 g/cm3)
- High hardness (HV10 ≥ 22 GPa)
- High flexibility (380- 430 MPa)
- High thermal conductivity (120-200 W/mK)
- Low linear expansion coefficient
- (3.6-4.1x10-6/K 20-400°C)
- SiC maximum operating temperature under shielding gas: 1800°C
- SiC excellent thermal shock resistance:ΔT 1100 K
- Corrosive
- Corrosion and wear resistance even at high temperatures
- Toxic safe
- Good sliding properties

The only compound of silicon and carbon is silicon carbide (SiC) or carborundum. SiC occurs naturally as the mineral moissanite, but this is extremely rare. However, it has been mass-produced in powder form for use as an abrasive since 1893. As an abrasive, it has been used for over a hundred years in grinding wheels and many other abrasive applications (Lisa K. Ross- n.d.)

SiC is a ceramic material with exceptional hardness surpassed only by diamond, cubic boron nitride and boron carbide. The material is abrasion resistant and chemically inert to all alkalis and acids as well as being high temperature resistant. These properties make Silicon Carbide a superior abrasive and ceramic material for use in extreme operating conditions. A representative image of Silicon Carbide is given in Figure 2.2.

Figure 2.2 Silicon carbide (Nanokar Website- n.d.)

2.1.3 Boron Carbide

Boron carbide (B4C) is a very hard boron-carbon ceramic and covalent material. The substance has many industrial applications, including tank armor, bulletproof vests, and engine sabotage powders. With a Vickers hardness of more than 30 HV, boron carbide is one of the hardest known materials, after cubic boron nitride and diamond. Boron carbide is coated on the surfaces of iron and alloyed metals for protection with the help of diffusion. In addition, it is much more advantageous to be used in heavy works and is very hard compared to other materials (Lucideon- 2001).

Various industrial areas where boron carbide is used;

- Metallurgy: Boron carbide is an important material in the metallurgical industry, thanks to its high temperature resistance, high hardness and chemical resistance properties. It is especially used in high temperature applications such as casting molds and hot knives.

- Defense Industry: Boron carbide is used in armor plates, tank armors and other defensive materials. Thanks to its high hardness and wear resistance, it is resistant to bullet piercing and explosive damage.

- Automotive Industry: Boron carbide is used in the automotive industry due to its temperature resistance and high hardness properties. It is especially used in the construction of parts that require wear resistance such as brake pads, fuel pumps and gearboxes.

- Electronics: Boron carbide, high temperature resistance, makes it very useful in the electronics industry due to its additional high hardness and electrical conductivity properties. In particular, it is used to make high-power semiconductors, high-frequency microchips and high-power LEDs.

- Mining: Boron carbide is used in the mining industry, especially in drilling equipment. Thanks to its high hardness, wear resistance and chemical resistance, it is used in the construction of drilling heads and other tools.

As a result, it is seen that boron carbide is used in many industrial applications especially due to its high temperature resistance, high hardness and wear resistance properties. It is an important material especially in the defense, automotive, metallurgy, electronics and mining industries. A representative image of Boron Carbide is given in Figure 2.3.

Figure 2.3 Boron carbide (Nanokar Website- n.d.)

2.1.4 Ethyl Silicate Resin

Polymeric materials resulting from the reaction of multifunctional acids and multifunctional alcohols are called polyester resins. The purpose of using the resin in our thesis is to use the ceramic powders mentioned above as binders in their mixtures.

We need a semi-fluid material to spray the coating on the ceramic fiber plate with a paint gun. For this, resin emerges as the most suitable component.

- It is used as a mold binder in the investment casting industry, as a component of fireproof paints and in the manufacture of high temperature resistant rods.
- It is used in the paint industry to obtain a stable gloss film that is resistant to high temperatures and water.
- It is used in the construction industry for the manufacture of water-repellent materials, for the treatment of painted levels, for reducing the pores of concrete, for the manufacture of silicon silicate and acid-resistant cement.
- In the glass industry it is used for the application of a light scattering layer on electric lamps, to increase the clarity of optical glasses.
- In the ceramic industry, it is used as a binder for ceramic paste, which is resistant to temperatures of 1750°C and pressure of 127kg/cm3, resistant to extremely aggressive environments and having high dielectric properties.

- In the textile sector it is used as a non-shrinking impregnation of wool fabrics, it is used as an impregnation for filter fabrics, which reduces the shrinkage of wool carpets and against rot and dust (Parashar, Srivastava, Kumar- 2001).

We have chosen the resin, whose properties are explained above, to be used at high temperatures for curing or heating processes. In this way, we wish the coating formed with high temperature ceramic powders to hold onto the ceramic fiber plate while preserving the binding property of the resin in the curing/heating section. A representative image of Ethyl Silicate Resin is given in Figure 2.4.

Figure 2.4 Ethyl silicate 40 resin (Zhonglan Industry- n.d.)

2.2 Mechanical and Chemical Properties of the Materials

Thanks to the materials we use, our main goal in the production of High Temperature and Friction Resistant Composite materials is;

- To increase the wear and friction resistance of the ceramic fiber plate thanks to the high temperature ceramic powders and resin we use,
- Based on the test results of the materials we use with each other. to perform a comparison,
- To increase heat resistance and reduce heat permeability.

Under this title, we will examine the mechanical properties of the materials we use.

It is important that we know the chemical and mechanical properties of the materials we use. Because in this way, we can prove how well the experimental results agree with them by making a comparison theoretically. The chemical and mechanical properties of the materials we use are given below. On the other hand, the ceramic fiber plate we used in our project is also used in the market as a high temperature resistant material. You can see examples of this in the figure... The High Temperature Ceramics that we will use as coatings on it have the same strength. Experimentally, our aim is to increase the resistance of the ceramic fiber plate against abrasion and friction, as well as to increase its resistance against high heat.

2.2.1 Ceramic Fiber Plate

Ceramic fiber plate is used as a material that stands out with its thermal absorption feature in high temperature furnaces, industrial furnaces, mechanisms operating at high temperatures and temperatures, such as boilers. Examples of this are shown below. In our study, a high-strength material that can be used in space and aviation will be used as the substrate, in other words, as the main material in the basic study phase. In Figure 2.5 and 2.6, sample pictures of the use of ceramic fiber board in ceramic kilns and as heat insulation material are given.

Figure 2.5 Usage in ceramic kilns (İzoref Website- n.d.)

Figure 2.6 Use as heat insulation material (Maghard Insulators Website- n.d.)

Table 2.1 Density heat resistance values of ceramic fiber plate

Usage Temperature **°C**	Sizes **Mm**	Plate Thickness **mm**	Density **Kg/m³**
1260-1430	1000 x 1200	10	300-360
1260-1430	1000 x 1200	25	300-360
1260-1430	1000 x 1200	50	300-360
1260-1430	1000 x 600	6	300-360
1260-1430	1000 x 600	9	300-360
1260-1430	1000 x 1200	100	300-360

The size of the heat tiles used in space vehicles is 15.2 cm on average, and the thickness varies according to the location used, and it is in the range of 2.54-12.7 cm. If a average weight of one heat tile calculation is made from here;

$$V=A_F \ x \ h \text{ (Prism Volume Formula)} \tag{2.1}$$

$$15.2 \times \left(\frac{2.54 + 12.7}{2}\right) \times 15.2 = 1760.5 \text{ cm}^3 = 0.0017605 \text{ m}^3$$

$$(1m^3 = 300kg) \Rightarrow 0.0017605 \ m^3 \Rightarrow 0{,}528 \text{ kg (m1)}$$

In this way, the weight of a composite material created with the materials we use in can be calculated approximately and on average.

However, heat tiles used in reality has a very low density and this value is approximately; 144 kg/m^3. If a calculation is made from here;

$$(1m^3 = 144kg) \Rightarrow 0.0017605 \text{ m}^3 \Rightarrow 0.253512 \text{ kg (m2)}$$

is calculated as.

If a comparison is made between two values;

$$(m1/m2) \tag{2.2}$$

$$\frac{0{,}528}{0.253512} = 2.08$$

result arises.

The conclusion to be drawn from here;

- A low weight coating material will be obtained,
- The success rate by weight is 48%.

Ceramic fiber plate components can be listed as follows;

Table 2.2 Ceramic fiber plate chemical component ratios (Kiron- 2021)

Classification Temperature	**1260°**	**1430°**	**1600°**
Density (kg/m^2)	300	300	300
Fracture Strength (MPa)	0,5	0,5	0,6
Thermal Shrinkage(W/m°K)	1,5	1,6	0,4
600°C		0,11	
800°C	0,22	0,15	0,15
1000°C		0,22	0,19
Chemical Composition	48	35	
Al_2O_3 %	51	44	
SiO_2 %		12 15	
ZrO_2 %			

Chemical Composition of Ceramic Fiber Plate

2.2.1.1 Aluminium Oxide

Aluminium oxide (or Aluminium (III) oxide) is a chemical compound of aluminium and oxygen with the chemical formula Al_2O_3. It is the most commonly occurring of several aluminium oxides, and specifically identified as aluminium oxide. Al_2O_3 is an electrical insulator but has a relatively high thermal conductivity ($30\ Wm^{-1}K^{-1}$) for a ceramic material. Aluminium oxide is insoluble in water.

Aluminum oxide mineral is formed due to the high sensitivity of aluminum metal to oxygen. They are preferred in the surface treatment sector due to their high hardness and high durability. (Wikipedia/ Aluminium Oxide- n.d.)

It is widely used in pipes and pipelines, flat glass and glass decoration processes, Teflon and pre-coating surface roughening, abrasive stone and sandpaper manufacturing, stone marble-granite cutting and refractory industry. A representative image of Aluminium Oxide is given in Figure 2.7.

Melting Point (°C):2050

Boiling Point (°C): 2990 (www.toppr.com/guides/physics/atomic-and-molecular-structure/aluminum-oxide-definition-uses-and-characteristics- n.d.)

Figure 2.7 Aluminium oxide (Nanokar Website- n.d.)

2.2.1.2 Silicon Dioxide

Silicon dioxide, also known as silica, is an oxide of silicon with the chemical formula SiO_2, most commonly found in nature as quartz. In many parts of the world, silica is the major constituent of sand. Silica is one of the most complex and most abundant families of materials, existing as a compound of several minerals and as a synthetic product.

Silicon dioxide is an anti-caking and moisture-retaining chemical component. Silicon dioxide is used in the pharmaceutical industry, paints and some powdered foods. It is also generally used for thickening and strengthening purposes.

Silicon dioxide is a highly insoluble, thermally stable silicon source suitable for glass, optics and ceramic applications. Oxide compounds do not conduct electricity and therefore are good insulators. However, certain perovskite structured oxides are the application of electronic conductor found at the cathode of solid oxide fuel cells and oxygen generation systems. Silicon dioxide amorphous films can be used as basic insulators in silicon integrated circuits, thanks to the above-mentioned properties. A representative image of Silicon Dioxide is given in Figure 2.8.

Melting point (°C): 1710

Boiling Point (°C): 2230 (Wikipedia/ Silicon Dioxide- n.d.)

Figure 2.8 Silicon dioxide (Nanokar Website- n.d.)

2.2.1.3. Zirconium Dioxide

Zirconium dioxide (ZrO^2) is a white crystalline oxide of zirconium. Of all the advanced ceramic materials, zirconia has the highest toughness and strength at room temperature Fracture toughness is essentially controlled by transformation toughening and is in the range 4–8 MPa√m depending on grain size, sintering temperature and yttria content. Regarding other mechanical properties, 3Y-TZP has high hardness and flexural strength generally around 1000 MPa, but can easily be even higher. Unlike other ceramic materials, zirconium dioxide (also known as zirconia) is a material that has a very high resistance to crack propagation. Zirconium dioxide ceramics also have very high thermal expansion and are therefore often the material of choice for joining ceramics and steel.

Another outstanding combination of properties is very low thermal conductivity and high strength. In addition, some types of zirconium dioxide ceramics can conduct oxygen ions. Components made from this material are significantly more expensive than components made from alumina ceramics. First used in the medical industry in 1969, zirconia has demonstrated outstanding biocompatibility with good tribological properties, good aesthetics and high mechanical properties.

Zirconium dioxide is highly resistant to cracking (including further development of cracks) and mechanical stress. Zirconium dioxide has a thermal conductivity of (2 $Wm^{-1}K^{-1}$), making it perfect for situations where heat needs to be controlled. (Wikipedia/ Zirconium Dioxide- n.d.)

This substance is chemically inert and non-reactive, working in industries that make use of various chemicals during processing. However, the compound is soluble in concentrated acids such as sulfuric or hydrofluoric acid. Zirconia's high mechanical properties, chemical inertness, high temperature stability, corrosion resistance and high quality have placed this ceramic material in many industries and applications. From refractories to medical products, pigments, electronics, coatings and ceramics, many of today's products are based on zirconia because of its superior properties and advantages over other materials. Some of the typical applications of zirconium dioxide include dies for hot metal extrusion, oxygen sensors, membranes in fuel cells, deep well valve seats and marine pump seals.

The mechanical strength and resistance of zirconium dioxide make it a suitable component for ceramic manufacture. This includes ceramic knives that are noticeably harder than steel-edged cutlery due to the zirconium dioxide high hardness factor. Because of its high thermal resistance, zirconium dioxide is used as a component in crucibles, furnaces and other high-temperature environments. In addition, zirconium dioxide increases the non-flammability properties of ceramics. Refractory bricks and armor plates are examples of zirconia-based refractory applications. Also, when fused quartz is added, zirconia can be used to produce siloxide glass, which is a tougher and more stress-resistant glass than quartz opaque glass. Zirconia can also be added to aluminum oxide for use in components for the steel casting process. Zirconium dioxide, it is applied as a coating to jet engine components exposed to high temperatures. This is made possible by the compound's low thermal conductivity and high heat resistance. Studies have confirmed the effectiveness of zirconium dioxide for TBC (Thermal Barrier Coating) applications as long as the material is applied uniformly and homogeneously. A representative image of Zirconium Dioxide is given in Figure 2.9.

Melting Point (°C): 2715

Boiling Point (°C): 4300 (Wikipedia/ Zirconium Dioxide- n.d.)

Figure 2.9 Zirconium dioxide (Nanokar Website- n.d.)

As a result, the common properties of the materials given above with their explanations are;

- High thermal conductivity,
- High temperature resistance,
- High mechanical strength
- High hardness value,
- Abrasion resistance.

2.2.2 Silicon Carbide

Since it is a hard material, it prevents wear. Thanks to this feature, it is one of the High Temperature Ceramic powders that we want to use as the main material in our thesis. In high friction conditions, the ceramic ware that we will use as a plate will take its place as a coating material on the plate. In this way, it is planned to prevent the wear behavior that will arise from friction. It is planned that the surface of the ceramic fiber plate will become more resistant to such abrasions. In Table 2.3 of chemical and mechanical properties of silicon carbide is given.

Table 2.3 Chemical and mechanical properties of silicon carbide

Density	3,21 g/cm^3
Vickers Hardness	29 GPa
Coefficient of Thermal Expansion	5*10^{-6}/K
Thermal Conductivity	50-100 W/m K
Typical Temperature Resistance	1500°C in air, 2400°C in inert atmosphere
Specific Heat	750/ kg K

2.2.3 Boron Carbide

Boron carbide is among the ceramic powders that we will use, it is the ceramic powder that we have the highest expectation in terms of strength. As we mentioned above, it is an increasingly popular substance in the world and in our country, and its use in many fields (aviation, weapons, nuclear, etc.) has begun and continues. (Boron Carbide (n.d.). Retrieved May 19, 2023, from Wikipedia)

This ceramic powder that we will use in our study is a high temperature resistant ceramic powder formed as a result of the reaction of boric acid and some carbon source at 2400°C. Thanks to its high chemical reaction and high temperature resistance, it is one of the key materials in our thesis. In Table 2.4 of chemical and mechanical properties of boron carbide is given.

Table 2.4 Chemical and mechanical properties of boron carbide (Lucideon- 2001)

Density	2,52 g/cm^3
Hardness (100 g. Knopp)	2900-3580 Kgf/mm^2
Crystal Structure	Rombohedral
Melting Temperature	2450 °C
Thermal Expansion	5x10^{-6} $°c'$
Electrical Conductivity	140 Ω^{-1}m^{-1}

CHAPTER THREE

MATERIALS AND METHOD

3.1 Material Production Steps (Inspiration)

There is a youtube video of Martin Wilson, who works as NASA TPS Operation Manager, as the inspiration and the criteria of the construction phase of the product we developed and put into the testing phase in our thesis. Since the basic construction stages, as well as the production stages in the articles and thesis topics, are not specifically mentioned, this video and the information on NASA's page are used as the infrastructure in our thesis.

Below are the images of the production stages of the material. In these figures, it is observed how a heat tiles are produced and what stages it goes through. In short, the most basic of the process is the conversion from a bulk material to a heat tiles. The materials used during this transformation and the visuals of the materials are shown in between Figures 3.1 and 3.14. (NASA 360 Thermal Protection System Tiles Space Shuttle. Retrieved May 24, 2021, from Youtube)

Figure 3.1 Martin Wilson, who works as NASA TPS Operation Manager (Personal Archive- 2023)

In Figure 3.2 it is emphasized that the main material, which is actually large in volume, is low in weight.

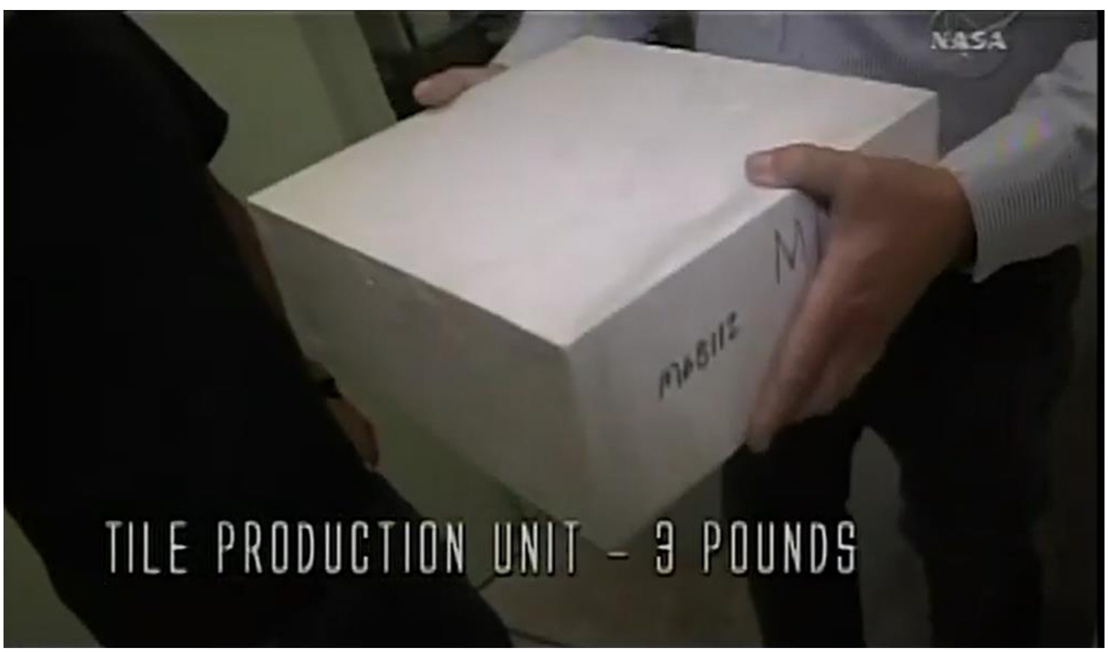

Figure 3.2 Tile production unit, 1.36078 kg (Personal Archive- 2023)

As seen in Figure 3.3, unlike our project, ceramic bulk is used in the actual production method.

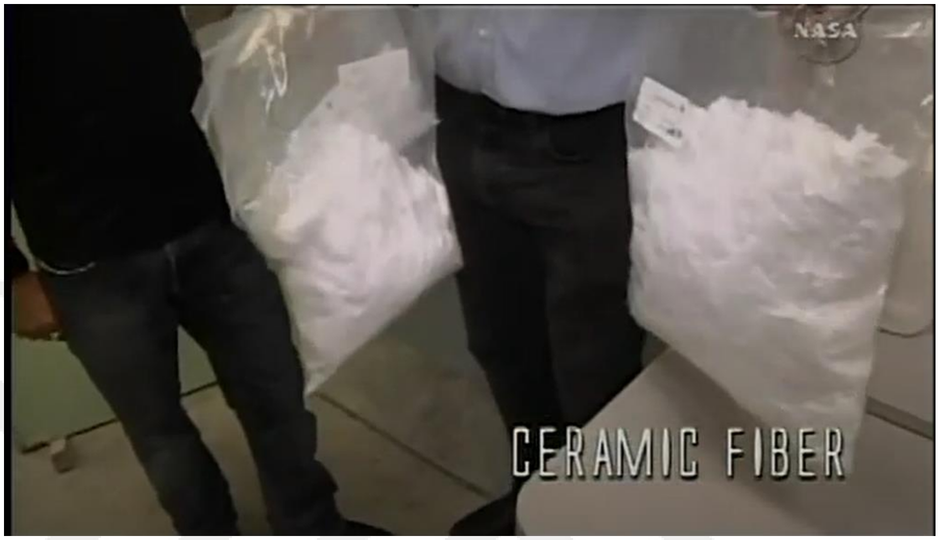

Figure 3.3 Ceramic fiber (bulk) (Personal Archive- 2023)

In Figure 3.4, the image of the mixture of high chemical content liquid and ceramic bulk is given.

Figure 3.4 Blender for chemical mixture, ceramic bulk and 25 gallon water mixing (Personal Archive- 2023)

Figure 3.5 shows the casting tower setup used for molding the mixture.

Figure 3.5 Casting tower (Personal Archive- 2023)

In Figures 3.6 and 3.7, a visual showing that 1 block of heat tiles is formed from 25 gallons of mixture material is given.

Figure 3.6 25 Gallons water to produce 1 block (heat tile) (Personal Archive- 2023)

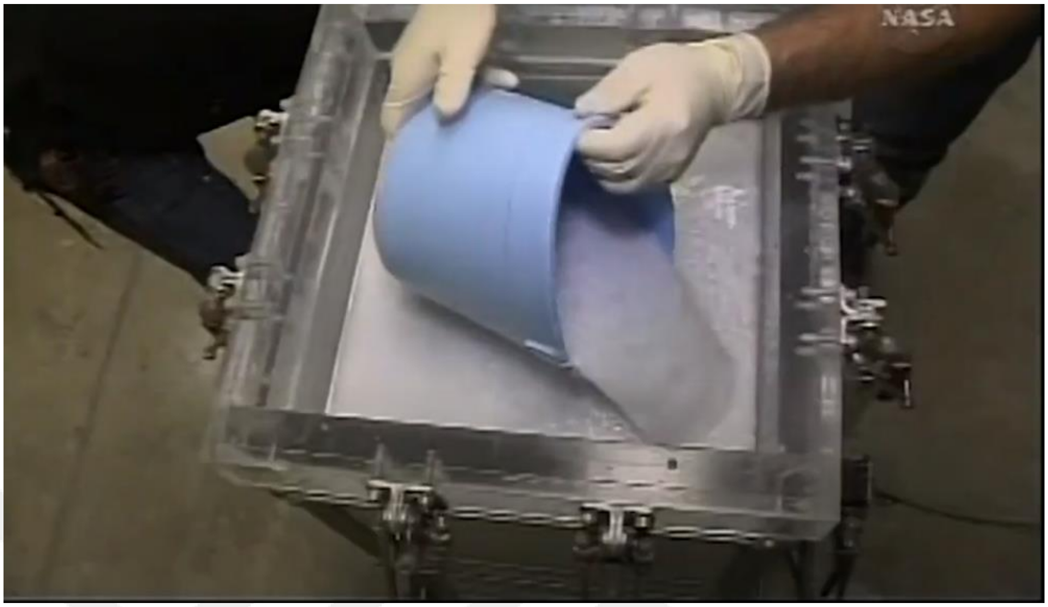

Figure 3.7 When adding mixture to the pressurized compression cabinet (Personal Archive- 2023)

In Figure 3.8, an image of how the excess chemical liquid in this 25 gallon mixture is discharged is given.

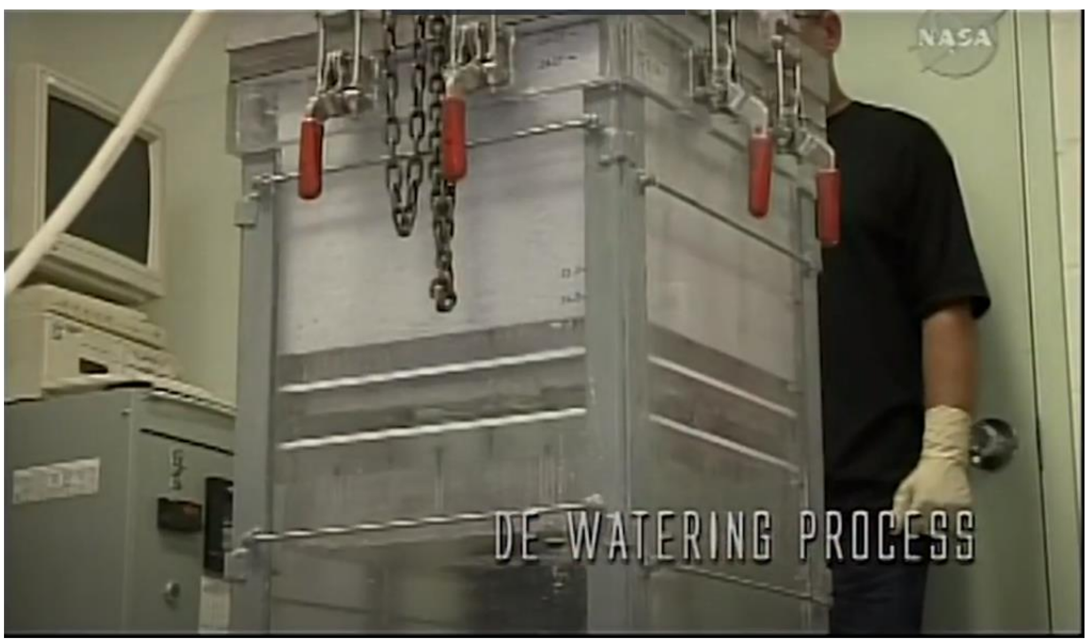

Figure 3.8 De-watering process (Personal Archive- 2023)

In Figure 3.9, the excess chemical liquid in this 25-gallon mixture is discharged. When this excess liquid is thrown out, a lighter and compressed heat tiles block is obtained.

Figure 3.9 De-Watering Process (When Draining Excess Mixture) (Personal Archive- 2023)

Figure 3.10 One block heat tile main material (Personal Archive- 2023)

Figure 3.11 shows the coating process on the base material. Here is the point that we need to pay attention and focus on in our thesis.

Figure 3.11 Coated heat tile (Personal Archive- 2023)

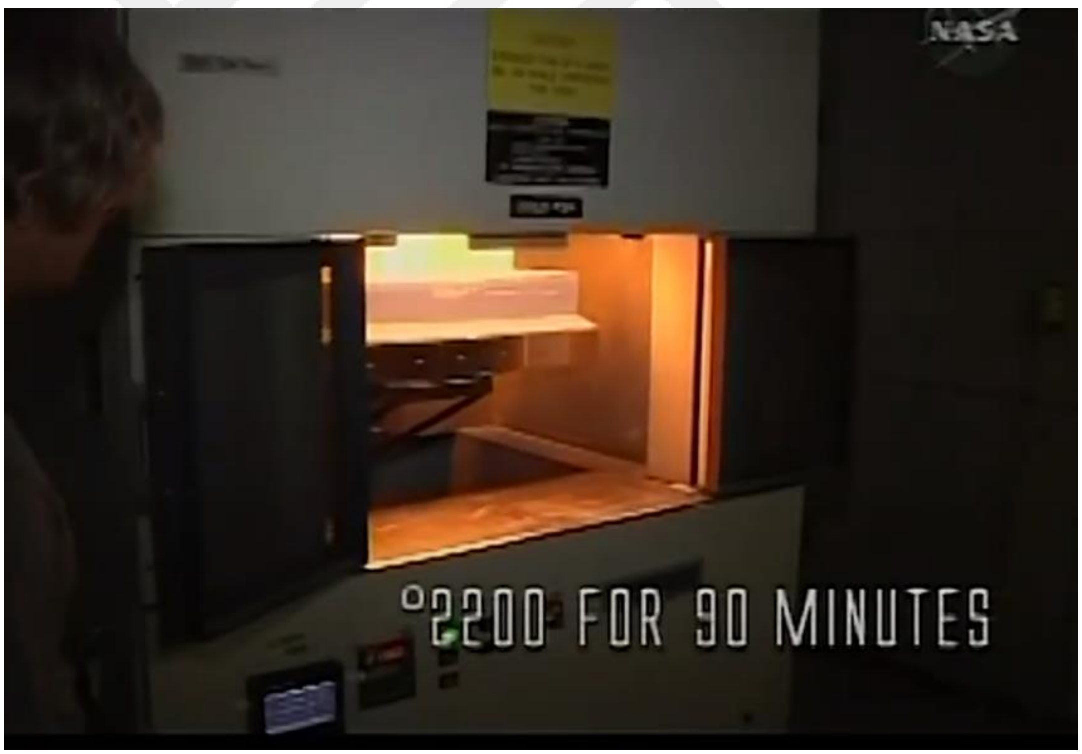

Figure 3.12 Heat treatment of heat tiles (Personal Archive- 2023)

Figure 3.13 shows the last material, namely heat tile. One of the most important points here is that this material is very light, has high surface strength and cools quickly. The point we will examine in our thesis is to examine the surface strength in terms of friction coefficient.

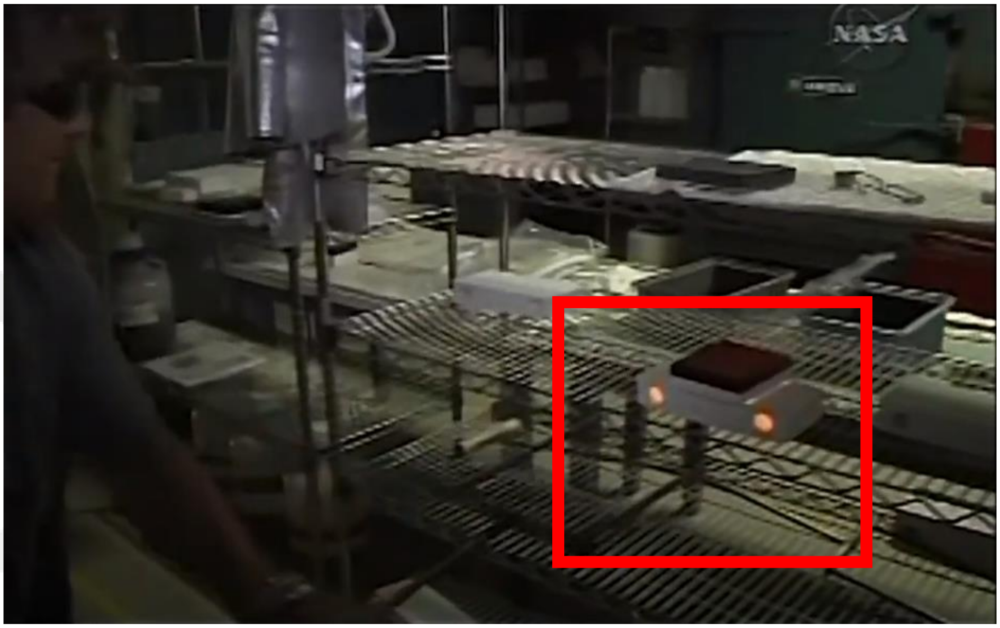

Figure 3.13 Heat treatment of heat tiles (Personal Archive- 2023)

Figure 3.14 Placement of heat tiles on spacecraft (Personal Archive- 2023)

Within the framework of the results obtained from here, we can list the production journey of a heat ceramic as follows;

- Ceramic Bulk-Fiber, which is preferred because of its lightness, is used as the infrastructure material.
- The Ceramic Fiber used with a chemical mixture in a blender rotated 360 degrees.
- The prepared fiber mixture is discharge to Casting Tower with 25 Gallons of water.
- The semi-liquid-fluid material obtained is taken into the De-Watering mold by means of the pressurized system.
- The resulting block is then taken to the processing benches and shaped according to the location where it will be placed in the spacecraft.
- The coating liquid is sprayed on the obtained Heat Tiles manually with a pressure paint spraying apparatus.
- The material is left to dry.
- Next it is kept in ovens at 2200℃ for 90 minutes.
- The product is subjected to test stages in a ready state.

3.2 Project Material Production

The visual image of the Heat Tiles that we produce is given in Figure 3.15 below in detail.

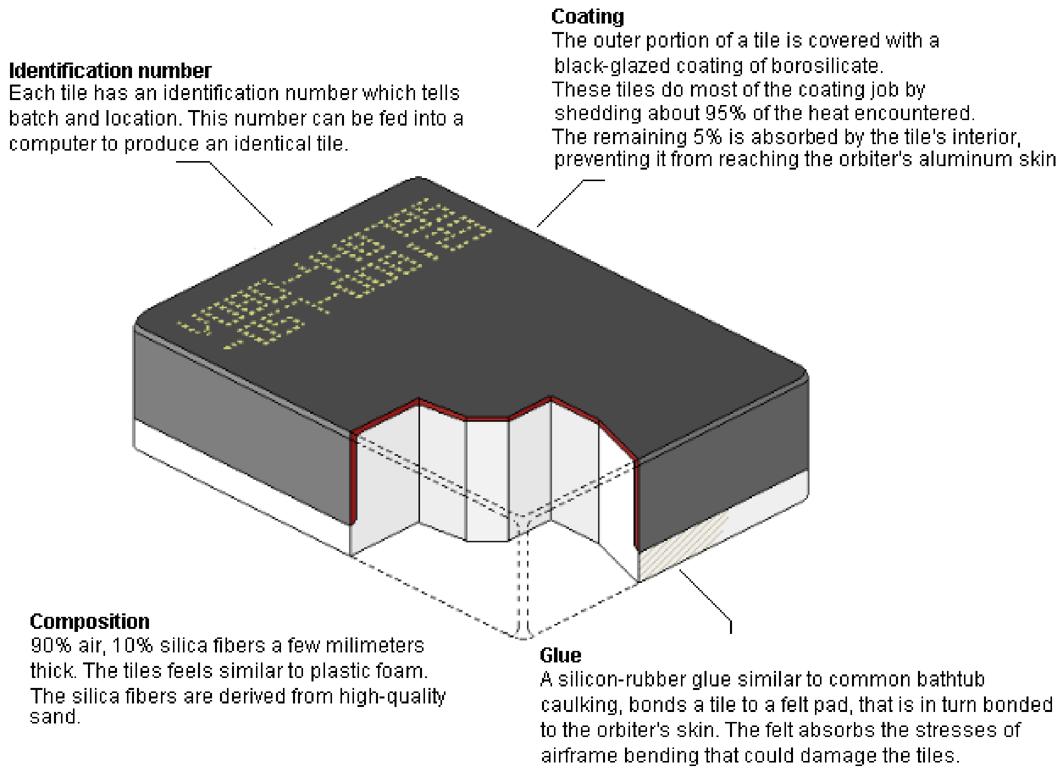

Figure 3.15 Heat tiles internal and external structure (Luis María Benítez- 2007)

Based on the figure above and the information we have mentioned before, the following steps have been followed to produce a coating material with high dynamic friction resistance on a high temperature resistant base.

The steps listed below were followed during the production phase of our project material, and these steps are detailed below in sequence with their visual images and explanations.

- Formation of the coating mixture.
- Laying out of the coating mixture on the substrate.
- Drying and heating process.

3.2.1 Formation of the Coating Mixture

The coating mixture basically consists of a combination of two materials. These two materials are High Temperature Ceramic Powder and Ethyl Silicate Resin. These two materials were mixed in a certain ratio (visually optimized mixture amount was determined as a result of various visual experiments as fluidity and coating quality) with a Magnetic Stirrer with heating setup. In the next step, the coating materials formed in this way were sprayed with the help of compressed air on to the surface to be coated with the help of a paint gun. The mixing amounts of the mixture made are given.

As mixing amount;

150 ml Ethyl Silicate Resin

25 gr Silicon Carbide Powder

Mixing Conditions

20°C / 25°C - 1000rpm / 2200rpm (The average speed was increased by 80rpm every minute.)

In Figure 3.16, a photograph depicting the preparation phase of the the mixture is given.

Figure 3.16 Preparation of the ethyl silicate resin and silicon carbide powder mixture (Personal Archive-2023)

Boron Carbide and Ethyl Silicate Resin come next as the second coating material mixture. The amount and conditions of mixing made here are given below.

As mixing amount;

150 ml Ethyl Silicate Resin

25 gr Boron Carbide Powder

Mixing Conditions

20°C / 25°C - 1000rpm / 2200rpm (The average speed was increased by 80rpm every minute.)

In Figure 3.17, a photograph depicting the preparation phase of the mixture is given.

Figure 3.17 Preparation of the ethyl silicate resin and boron carbide powder mixture (Personal Archive-2023)

Boron Carbide, Silicon Carbide and Ethyl Silicate Resin come next as the third coating material mixture. The amount and conditions of mixing made here are given.

As mixing amount;

150 ml Ethyl Silicate Resin

12,5 gr Boron Carbide Powder

12,5 gr Silicon Carbide Powder

Mixing Conditions

20°C / 25°C - 1000rpm / 2200rpm (The average speed was increased by 80rpm every minute.)

In Figure 3.18, shows the preparation of the mixture.

Figure 3.18 Preparation of the ethyl silicate resin & powder mixture (Personal Archive- 2023)

The mixtures formed in this way were mixed in a Magnetic Stirrer with Heating for 15 minutes at certain rpm ranges and temperatures, as stated above. Magnetic Stirrer with heating setup is a device that allows the liquids in glass containers that can be placed on its large circular table to be mixed with the effect of the magnetic field created and heated by the heaters in the tray. To understand the magnetic stirrer device, the following example can be given: The sugar added to a glass of tea can be mixed more quickly and homogeneously if the tea is hot enough and mixed. A representative image of Magnetic Stirrer with Heating is given in Figure 3.19.

Figure 3.19 Magnetic stirrer with heating (CarlRoth Website- n.d.)

A magnetic fish is used to create a magnetic field. In order to mix the solution in the beaker homogeneously, the magnetic fish is thrown into it. The magnetic fish is in the form of iron balls. The magnet head rotating together with the motor in the magnetic stirrer allows these magnetic fishes to rotate in the mixture. The ultramagnetic fishes are cylindrical in shape and have rounded ends. It is produced by coating the cylindrical magnet inside with polymer. Because of its shape, its usage area is very wide. The polymer sheath is heat resistant up to +270 °C. In Figure 3.20, the visual of the magnetic fish is given. (T.C. Milli Eğitim Bakanlığı Biyomedikal Cihaz Teknolojileri, Manyetik Karıştırıcı Cihazları- 2011)

Figure 3.20 Magnetic fish (Fisher Scientific Website- n.d.)

3.2.2 Coating of The Coating Mixture on The Substrate

The coating process was carried out with an Air Spray Gun. Cylindrical pieces of approximately 30mm in diameter and 15mm in height cut from ceramic fiber plates were placed outdoors. The coating process was done outdoors to prevent health problems.

A sprayer is a tool used to apply paint. This tool, which usually works with an air compressor, is used to spray paint and other materials with the help of air. Spray guns are used in the application of various paints and painting in large areas. These tools can work more quickly and effectively than applying paint by hand.

The coating mixture made on the ceramic fiber plate was evenly distributed by eye. An average of 1 mm was determined as the coating thickness. Images of the process performed are given in Figures 3.21, 3.22 and 3. 23.

Figure 3.21 Boron carbide coating sample (Personal Archive- 2023)

Figure 3.22 Boron carbide & silicon carbide coating sample (Personal Archive- 2023)

Figure 3.23 Silicon carbide coating sample (Personal Archive- 2023)

3.2.3 The Drying and Heating Process

The samples prepared with the methods mentioned above are hardened first by drying and then by heating, respectively. The main purpose here is to increase the mechanical strength of the material by providing the hardening of the coating material.

First, the materials were left to dry in room conditions in a period of 24 hours. The surfaces of the drying materials (especially in the silicon carbide coating sample a large amount of moisture is observed) still maintain moderate moisture. Since the materials cannot be introduced into the tribometer in this way, the next step, the heating process, must be started.

The first method we tried for the heating process was the ceramic curing oven (Dokuz Eylul University Materials and Metallurgical Engineering Ceramic Curing Furnace). In the first trial, after drying the coated ceramic fiber plate sample in an oven for 60 minutes, it was left to heat-cure at 1000°C for 90 minutes (as in the heating method used in NASA). However, after the material was removed from the furnace, it was burnt and was almost in completely ash form. Ceramic fiber plate was almost similar in appearance to concrete material.

As the next experiment, the sample formed with the same coating and materials in the same oven was heated at 300°C for 30 minutes. As a result, extreme crater cracks were observed on the surface this time.

After these unsuccessful attempts, the agreed method for heating was the domestic oven. The average heating time of 15 minutes in a household oven up to 240°C was determined to be sufficient. The images of the samples obtained afterwards are shown in

Figures 3.24 & 3.25 & 3.26 (left to right; boron carbide, boron carbide& silicon carbide, silicon carbide coating).

Figure 3.24 Image of parameter changes in a household oven (Personal Archive- 2023)

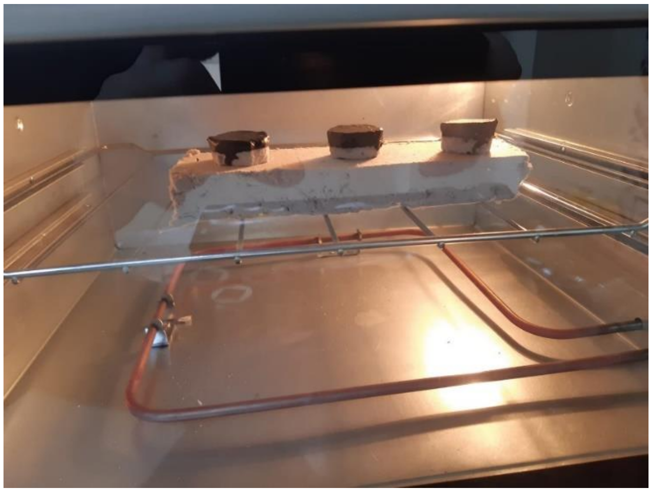

Figure 3.25 Heating process in a household oven (Personal Archive- 2023)

Figure 3.26 Samples after heating process is completed (Personal Archive- 2023)

Except for the samples prepared for the tribometer test setup above, samples of approximately 150x150x25 mm were prepared, such as the molds used in spacecraft. The visual of the samples is shown in Figure3.27(left to right; boron carbide, boron carbide & silicon carbide, silicon carbide coating).

Figure 3.27 Visual of the samples (Personal Archive- 2023)

The differences and observed features between the samples we prepared above are as follows;

- Superficial cracks are observed in the Boron Carbide coating even before the heating process is started.
- The heating process of Silicon Carbide coating took longer than other materials (approximately 2 times). The reason is that in the mixture of resin and silicon carbide, the adhesion of the resin to the material surface was less than in other mixtures. And for this reason, its volatility was much higher compared to the other 2 samples.
- Boron carbide coating was the hardest material visually in terms of surface coating.
- The resin showed almost no volatility in the boron carbide coating. The coating is fully adapted to the material.

3.3 Dynamic Friction Coefficient

Samples prepared as 30(Ø)x15 mm were tested at Celal Bayar University Tribometer laboratory.

The visuals of the test pieces are shown in Figure 3.28 (top left to right (boron carbide & boron carbide mixture, boron carbide coating) down left to right (silicon carbide coating, uncoated base material).

Figure 3.28 Test pieces prepared (Personal Archive- 2023)

As can be seen in the figure, in addition to the previously mentioned 3 different coating materials, 1 uncoated raw material (ceramic fiber plate) was included in the test phase. In this way, we can learn how much a material without a coating has been improved in terms of dynamic friction.

However, since the prepared materials could not adapt to the tribometer device (the holders could not grasp a round object due to the low weight), the edges were cut and turned into the shape shown in Figure 3.29.

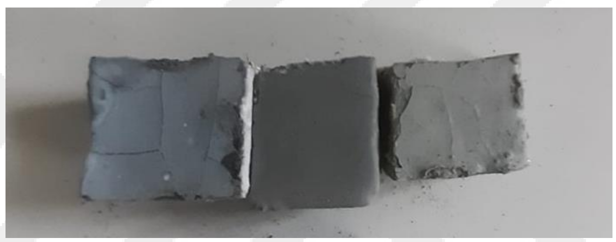

Figure 3.29 Samples prepared for tribometer (Personal Archive- 2023)

The prepared samples were placed in the tribometer device. The operating system of the tribometer device is connected to a computer. The amount of weight on which dynamic friction is applied affects the ball on disc mechanism in Newtons. The photograph of the system prepared below is shown in Figure 3.30 & Figure 3.31.

Figure 3.30 Tribometer (Personal Archive- 2023)

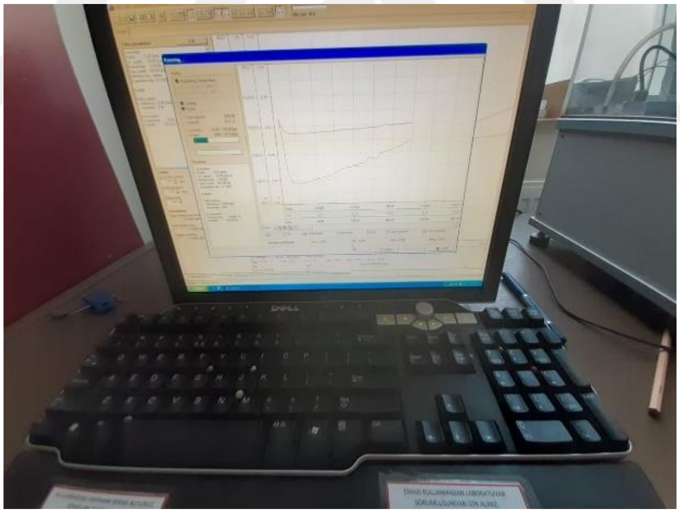

Figure 3.31 Data acquisition of the tribometer by computer (Personal Archive- 2023)

In addition to the visuals above, the visual recorded during the study is shown in Figure 3.32.

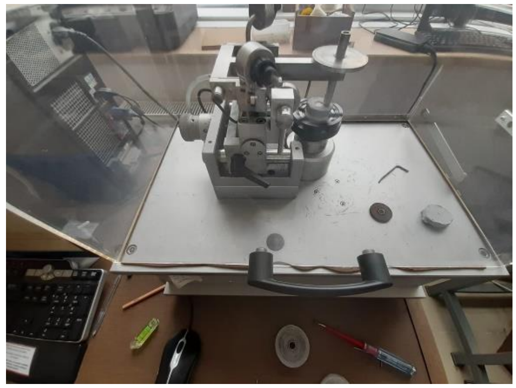

Figure 3.32 Visual recorded during the study (Personal Archive- 2023)

As a result of the tribological experiment, a friction field was created on the samples with the circular pressure method. The conditions created in this experiment are given in Table 3.1.

Table 3.1 Tribological parameters used in the experiments

Acquisition Radius (mm)	Linear speed [cm/s]	Normal load [N]	Acquisition rate [hz]
5.01	10.00	1.00	0.1

The final state of the materials as a result of the above-mentioned tribological experiment is shown in Figure 3.33 & Figure 3.34 & Figure 3.35 & Figure 3.36.

Figure 3.33 Test result of uncoated raw material (image) (Personal Archive- 2023)

Figure 3.34 Test result of silicon carbide coated material (image) (Personal Archive- 2023)

Figure 3.35 Test result of silicon carbide & boron carbide coated material (image) (Personal Archive- 2023)

Figure 3.36 Test result of boron carbide coated material (image) (Personal Archive- 2023)

As can be seen from the photographs given above, the damage size of the material showed a decreasing trend from Figure 3.33 to Figure 3.36. In other words, based on the above images, we can say that the silicon carbide coating showed better surface strength compared to the uncoated material, the mixture coating showed better surface strength compared to the silicon carbide coating, and the boron carbide coating showed better surface strength compared to the mixed coating.

In Figure 3.37, uncoated material, silicon carbide coating and boron carbide coating are shown side by side for a better visual comparison.

Figure 3.37 Visual comparison of uncoated and coated materials (Personal Archive- 2023)

CHAPTER FOUR

RESULTS AND DISCUSSION

Under this heading, we will include the results and inferences we have reached as a result of the above-mentioned material production and tests.

The inputs of the tribometer and environment conditions are given in Table 4.1below.

Table 4.1 The inputs of the tribometer and environment conditions

Radius	Linear Speed	Normal Load	Stop Condition	Effective Stop	Acquisition Rate	Dimension	Geometry	Tempretaure	Humidity
5.01 [mm]	10.00 [cm/s]	1.00 [N]	100.00 [m]	Meters	0.1 [hz]	6.00 [mm]	Ball	20.00 [°C]	50.00 [%]

Coefficient of friction, ratio of the frictional force resisting the motion of two surfaces in contact to the normal force pressing the two surfaces together. It is usually symbolized by the Greek letter mu (μ). Mathematically, $\mu = F/N$, where F is the frictional force and N is the normal force. Because both F and N are measured in units of force (such as newtons or pounds), the coefficient of friction is dimensionless.

The tribological start, minimum, maximum and mean (μ) results of the ceramic fiber plate, whith no coating, are given in Table 4.2.

Table 4.2 The μ results of the ceramic fiber plate

Start (μ)	Minimum(μ)	Maximum(μ)	Mean(μ)
0.78538	0.67924	1.02879	0.93048

The μ distributions for ceramic fiber plate by time are given in Table 4.3 below.

Table 4.3 The μ distributions for ceramic fiber plate by time

Time (s)	Distance (m)	Laps	μ
10	0.975	31	0.78539
20	1.981	63	0.86302
30	2.956	94	0.87573
40	3.963	126	0.87414
50	4.969	158	0.86065
60	5.976	190	0.87450
70	6.983	222	0.88840
80	7.979	253	0.88489
90	8.981	285	0.94379
100	9.983	317	0.93221
110	10.985	349	0.92329
120	11.988	380	0.91922
130	12.990	412	0.92028
140	13.992	444	0.91816
150	14.994	476	0.93972
160	15.996	507	0.96073
170	16.998	539	0.94757
180	18.001	571	0.95005
190	19.003	603	0.95702
200	20.005	634	0.97827
210	21.007	666	0.96914
220	22.009	698	0.96359
230	23.012	730	0.97353
240	24.014	762	0.96982
250	25.016	793	0.96542
260	26.018	825	0.98943
270	27.020	857	1.00703

Table 4.3 continued

280	28.022	889	1.02081
290	29.025	920	1.01693
300	30.027	952	1.02880
310	31.029	984	1.02117
320	32.031	1016	1.00696
330	33.033	1047	0.82771
340	34.035	1079	0.67924
350	35.038	1111	0.16053
360	36.040	1143	0.07371
370	37.042	1174	0.04306
380	38.044	1206	0.01371
390	39.046	1238	-0.01633
400	40.048	1270	-0.03955
410	41.051	1301	-0.04661
420	42.053	1333	-0.05417
430	43.055	1365	-0.06401
440	44.057	1397	-0.04872
450	45.059	1429	-0.07061
460	46.061	1460	-0.08082
470	47.064	1492	-0.08797
480	48.066	1524	-0.08832
490	49.068	1556	-0.09386
500	50.070	1587	-0.09550
510	51.072	1619	-0.10173
520	52.074	1651	-0.10715
530	53.077	1683	-0.11000
540	54.079	1714	-0.11393
550	55.081	1746	-0.11618
560	56.083	1778	-0.12088
570	57.085	1810	-0.12145
580	58.088	1841	-0.12325
590	59.090	1873	-0.12474
600	60.092	1905	-0.12632

The tribological start, minimum, maximum and mean (μ) results of the ceramic fiber plate, with silicon carbide coating, are given in Table 4.4.

Table 4.4 The μ results of the silicon carbide coating

Start (μ)	Minimum(μ)	Maximum(μ)	Mean(μ)
0.58205	0.57844	1.05478	0.74929

The μ distributions for Silicon Carbide Coating by time are given in Table 4.5.

Table 4.5 The μ distributions for silicon carbide coating by time

Time (s)	Distance (m)	Laps	μ
10	0.97506	31	0.58206
20	1.981	63	0.64989
30	2.956	94	0.66000
40	3.963	126	0.67649
50	4.969	158	0.66989
60	5.976	190	0.65704
70	6.982	222	0.64984
80	7.957	253	0.63965
90	8.964	285	0.63240
100	9.970	317	0.62733
110	10.977	349	0.62803
120	11.952	380	0.63052
130	12.964	412	0.63316
140	13.964	444	0.63141
150	14.963	476	0.63453
160	15.962	508	0.63801
170	16.962	539	0.64123
180	17.961	571	0.64185
190	18.960	603	0.63808
200	19.960	635	0.63585
210	20.959	667	0.63826
220	21.959	698	0.64757
230	22.958	730	0.64850
240	23.957	762	0.64831
250	24.957	794	0.65284
260	25.956	825	0.65174
270	26.955	857	0.65096

Table 4.5 continued

280	27.955	889	0.66552
290	28.954	921	0.66921
300	29.953	953	0.67916
310	30.953	984	0.68416
320	31.952	1.016	0.69526
330	32.952	1.048	0.70307
340	33.951	1.080	0.70734
350	34.950	1.111	0.72506
360	35.950	1.143	0.73778
370	36.949	1.175	0.76576
380	37.948	1.207	0.78933
390	38.948	1.239	0.81143
400	39.947	1.270	0.82752
410	40.947	1.302	0.93183
420	41.946	1.334	1.02618
430	42.945	1.366	0.97835
440	43.945	1.398	1.05475
450	44.944	1.429	0.97504
460	45.943	1.461	0.92126
470	46.943	1.493	0.92689
480	47.942	1.525	0.91166
490	48.942	1.556	0.91619
500	49.941	1.588	0.94024
510	50.940	1.620	0.94408
520	51.940	1.652	0.96637
530	52.939	1.684	0.98091
540	53.938	1.715	0.98300
550	54.938	1.747	0.99072
560	55.937	1.779	0.92883
570	56.937	1.811	0.88238
580	57.936	1.843	0.76059
590	58.935	1.874	0.64472
600	59.935	1.906	0.57844

The tribological start, minimum, maximum and mean (μ) results of the silicon carbide & boron carbide coating are given in Table 4.6.

Table 4.6 The μ results of the silicon carbide & boron carbide coating

Start (μ)	Minimum(μ)	Maximum(μ)	Mean(μ)
0.53045	0.43457	0.56859	0.49563

The μ distributions for Silicon Carbide & Boron Carbide Coating by time are given in Table 4.7.

Table 4.7 The μ distributions for silicon carbide & boron carbide coating by time

Time (s)	Distance (m)	Laps	μ
10	0.9750	31	0.53046
20	1.981	63	0.45686
30	2.988	95	0.43652
40	3.963	126	0.43584
50	4.969	158	0.43457
60	5.976	190	0.43578
70	6.982	222	0.43681
80	7.957	253	0.43751
90	8.964	285	0.44070
100	9.970	317	0.44245
110	10.977	349	0.44345
120	11.983	380	0.44485
130	12.958	412	0.44528
140	13.967	444	0.45064
150	14.966	476	0.45284
160	15.965	508	0.45600
170	16.964	539	0.45807
180	17.963	571	0.45802
190	18.962	603	0.46151
200	19.961	635	0.46132
210	20.960	666	0.46481
220	21.959	698	0.46481
230	22.958	730	0.47262
240	23.958	762	0.47194

Table 4.7 continued

250	24.957	793	0.47078
260	25.956	825	0.47929
270	26.955	857	0.48175
280	27.954	889	0.48282
290	28.953	920	0.48386
300	29.952	952	0.48781
310	30.951	984	0.48965
320	31.950	1.016	0.49408
330	32.950	1.048	0.49720
340	33.949	1.079	0.49803
350	34.948	1.111	0.50062
360	35.947	1.143	0.50328
370	36.946	1.175	0.50817
380	37.945	1.206	0.51395
390	38.944	1.238	0.51533
400	39.943	1.270	0.51832
410	40.942	1.302	0.52042
420	41.941	1.333	0.52570
430	42.941	1.365	0.53069
440	43.940	1.397	0.53280
450	44.939	1.429	0.53416
460	45.938	1.460	0.53694
470	46.937	1.492	0.53882
480	47.936	1.524	0.53816
490	48.935	1.556	0.54004
500	49.934	1.588	0.54375
510	50.933	1.619	0.54778
520	51.933	1.651	0.54746
530	52.932	1.683	0.55188
540	53.931	1.715	0.55449
550	54.930	1.746	0.55660
560	55.929	1.778	0.55980
570	56.928	1.810	0.56136
580	57.927	1.842	0.56319
590	58.926	1.873	0.56687
600	59.925	1.905	0.56859

The tribological start, minimum, maximum and mean (μ) results of the boron carbide coating are given in Table 4.8.

Table 4.8 The μ results of the boron carbide coating

Start (μ)	**Minimum(μ)**	**Maximum(μ)**	**Mean(μ)**
0.17241	0.16497	0.23898	0.20978

The μ distributions for Boron Carbide Coating by time are given in Table 4.9 below.

Table 4.9 The μ distributions for boron carbide coating by time

Time (s)	**Distance (m)**	**Laps**	**μ**
10	0.9750	31	0.17241
20	1.981	63	0.16526
30	2.988	95	0.16497
40	3.963	126	0.16833
50	4.969	158	0.17077
60	5.976	190	0.17331
70	6.982	222	0.17672
80	7.957	253	0.17866
90	8.964	285	0.18101
100	9.970	317	0.18251
110	10.977	349	0.18575
120	11.983	380	0.18803
130	12.971	412	0.18922
140	13.971	444	0.19004
150	14.971	475	0.19328
160	15.970	507	0.19455
170	16.970	539	0.19689
180	17.969	571	0.19940
190	18.969	602	0.20056
200	19.969	634	0.20199

Table 4.9 continued

210	20.968	666	0.20378
220	21.968	698	0.20494
230	22.968	729	0.20538
240	23.967	761	0.20557
250	24.967	793	0.20785
260	25.966	825	0.21020
270	26.966	856	0.21096
280	27.966	888	0.20944
290	28.965	920	0.21017
300	29.965	951	0.21265
310	30.964	983	0.21427
320	31.964	1.015	0.21583
330	32.964	1.047	0.21550
340	33.963	1.078	0.21615
350	34.963	1.110	0.21721
360	35.963	1.142	0.21884
370	36.962	1.174	0.22094
380	37.962	1.205	0.22227
390	38.961	1.237	0.22188
400	39.961	1.269	0.22234
410	40.961	1.301	0.22320
420	41.960	1.332	0.22572
430	42.960	1.364	0.22646
440	43.960	1.396	0.22767
450	44.959	1.427	0.22991
460	45.959	1.459	0.23125
470	46.958	1.491	0.23081
480	47.958	1.523	0.23164
490	48.958	1.554	0.23188
500	49.957	1.586	0.23262
510	50.957	1.618	0.23529
520	51.956	1.650	0.23226
530	52.956	1.681	0.23185
540	53.956	1.713	0.23368
550	54.955	1.745	0.23493
560	55.955	1.777	0.23585
570	56.955	1.808	0.23703

580	57.954	1.840	0.23898
590	58.954	1.872	0.23773
600	59.953	1.903	0.23865

As seen in the tables above, the order of friction coefficients is;

Boron Carbide < Silicon Carbide & Boron Carbide < Silicon Carbide < Ceramic Fiber.

The starting, minimum, maximum and mean values are shown together in Table 4.10.

Table 4.10 The starting, minimum, maximum and mean values

Ceramic Fiber	**Start (μ)**	**Minimum(μ)**	**Maximum(μ)**	**Mean(μ)**
	0.78538	0.67924	1.02879	0.93048
Silicon Carbide	**Start (μ)**	**Minimum(μ)**	**Maximum(μ)**	**Mean(μ)**
	0.58205	0.57844	1.0547	0.74929
Silicon Carbide & Boron Carbide	**Start (μ)**	**Minimum(μ)**	**Maximum(μ)**	**Mean(μ)**
	0.53045	0.43457	0.56859	0.49563
Boron Carbide	**Start (μ)**	**Minimum(μ)**	**Maximum(μ)**	**Mean(μ)**
	0.17241	0.16497	0.23898	0.20978

As is known, the formula for calculating the friction force is as follows;

$$Fs\ (\text{Newton}) = \mu \ .\ N\ (\text{Newton}) \qquad (4.1)$$

That is, the higher the coefficient of friction, the greater the friction force. In short, the coefficient of friction is directly proportional to the friction force.

$$E\text{ısı}\ (\text{Joule}) = W(\text{Joule}) = Fs\ (\text{Newton})\ .\ Dx\ (\text{Meter}) \qquad (4.2)$$

As stated in 4.2, heat energy in a system is equal to the product of the frictional force and the distance traveled. In other words, the heat energy released in a system is directly proportional to the friction force between the moving objects and the distance traveled in that system.

$$E_{ISI} = F_s . Dx \;\; = \mu . N . Dx \qquad (4.3)$$

As it can be understood from 4.3, when the formulas given above are combined, the direct ratio between the friction coefficient and the heat energy released in a system emerges.

That is, the higher the coefficient of friction, the more the system will heat up. This is a negative effect for the heat tiles used in spacecraft, which is our purpose of departure. Therefore, the lower the friction coefficient of our coating material, the higher its efficiency will be.

In space vehicles, there is a high dynamic friction between the vehicle and the atmosphere. This dynamic friction has a negative impact on the vehicle in terms of high heat and consequently temperature. The more heat generated by friction, the higher the probability of wear and damage to the heat tiles. For these reasons, the coefficient of friction should be minimized.

Based on the tables given above, it turns out that the most suitable material for this process is Boron Carbide, which has a lower friction coefficient compared to other coatings. From this point of view, the contribution of Boron Carbide High Temperature Resistant Ceramic material to Silicon Carbide under dynamic friction conditions is revealed by comparing Tables 4.7 and 4.9.

CHAPTER FIVE

CONCLUSIONS AND FUTURE WORK

Based on these results, the following inferences can be made;

1. Boron Carbide coating has the lowest dynamic friction coefficient.
2. Boron Carbide, which has the lowest dynamic coefficient of friction, will result in the lowest temperature that will occur in the simulative display between the atmosphere and the spacecraft.
3. Thanks to the Boron Carbide additive, the Silicon Carbide coating has also been strengthened in terms of dynamic friction coefficient.
4. As seen in Table 4.3, in the uncoated ceramic fiber plate, the values suddenly decreased after 350 seconds and decreased to negative values after 390 seconds. This is because the material is so worn that the friction device cannot measure (Specified places are drawn with yellow marker).
5. Silicon Carbide coating also strengthened the material in terms of dynamic friction coefficient.
6. An important proof has been obtained for the Turkish Aerospace Industry, such as Boron Carbide, whose main material is the element Boron and that the majority of its reserves are in our country.

Our aim in our thesis was to produce a material similar to the heat tiles used in spacecraft and to analyze it based on the surface friction coefficient. In line with the results we obtained, it has been observed that successful results can be obtained with boron carbide coating. Boron carbide stands out as a material used in the field of aviation and space today. We see that successful results can be obtained if it is also used in the outer coatings of space vehicles.

If it is necessary to consider our thesis from another angle, the point we should focus on here is the outer coating design of spacecraft. The reason why this issue is so important is the fact that the most difficult moments during a spacecraft's journey are entering and exiting the atmosphere. If there is no problem at these moments, we can say that the trip was successful. Because the first purpose of a space and aviation trip is the successful flight and landing of the crew. Here, too, the most important task is to ensure that the heat generated as a result of atmospheric friction does not pass into the spacecraft. Here, too, the biggest task falls on the external insulation coatings. We believe that if this purpose is combined with boron carbide, which is the raw material of the boron mine, which is abundant in terms of reserves in our country, a good result and future will emerge.

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