Analysis of Ultra-High Temperature Ceramic Using CAD Tools for Aerospace Applications

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***Abstract*: Ceramic materials having melting points higher than 3000 °C and suitable for structural applications at above 2000°C are commonly known as Ultra-High Temperature Ceramics (UHTCs). Over the last couple of decades, there has been a growing interest for UHTCs in general, and for the transition metal di-borides in particular, due to the increasing demands in hypersonic aerospace vehicles, atmospheric re- entry vehicles and energy applications. However, problems pertaining to sintering, moderate fracture toughness and experimental challenges associated with reliably measuring the elevated temperature properties, as well as the properties that determine the performances at the actual service conditions, have limited their widespread applications. This paper comprehensively reviews the various routes/techniques, including the advanced ones, as adopted for the synthesis and densification of the di- borides. The effects of sinter-additives and reinforcements on the densification, microstructure and various properties, including elevated temperature properties have been discussed in critical terms. Due attention has been paid towards understanding the challenges associated with the experimental measurements of the high temperature properties under extreme environmental conditions and the very recently developed techniques for the same. Some of the existing and futuristic applications of transition metal di- borides have also been discussed. Finally, the review concludes with an outlook towards some of the outstanding issues.**

***Keywords*: Ceramic materials having melting points, Ultra- High Temperature Ceramics, The effects of sinter-additives and reinforcements**

INTRODUCTION

The space vehicles like rockets, hypersonic flights, reentry vehicles work at supersonic and hypersonic speeds (Mach number 6 or 7). At such high speeds leading and sharp edgesof space vehicles are subjected to very high aerodynamic heating than other blunt edges of the vehicle. During the reentry operation, they reach temperatures of 2000°C. Thus,to protect orbiter of the space shuttle, thermal protection system (TPS) is used. TPS of the orbiter of the space shuttle is designed to work effectively over an environment's spectrum typically for both aircraft and space shuttle. While designing TPS orbiter temperature must be maintained less than 177°C (350°F) [1]. Materials used for TPS should have high- temperature stability, high oxidation resistance, high corrosion resistance etc. Also, the TPS must perform acceptably in other environments, i.e., structural deflections induced by aerodynamic loads, on-orbit cold soak, and natural environments, such as salt, fog, wind, and rain. Selection and location of the various TPS materials used for orbiter structure mainly depends on its inherent temperature capability. The location of materials on orbiter of spacecraft mainly depends on predicted maximum surface temperature and its reuse temperature. These requirements can be fulfilledby ultra-high temperature ceramics (UHTC). UHTCs are a new class of materials that have the potential foruse in extreme environments. UHTCs are the compounds having melting points greater than 2000°C. Generally, all UHTCs are binary compounds which contain boron, carbon, or nitrogen combine transition metals (TMs) such as Zr, Hf, Ti, Nb and Ta. The strong covalent bond between the TMs and B, C, or N causes high hardness, stiffness, and melting temperature [5]. Currently, structure materials used for high- temperature oxidizing environment limited to silicon carbide or Si3N4 based oxides materials and C/C composite. Also, materials used must withstand in high heat Flux with heavy mechanical stresses. These materials exhibit better oxidation resistance only up to 1600°C and their thermal cycling lifetimes are modest. Therefore, the development of materialsfor use in oxidizing and rapid heating environments at a temperature above 1600°C is of great engineering significance. Hence to fulfil such high-temperature structural applications like hypersonic space vehicles, propulsion component, furnace elements and refractory crucibles etc. ultra-high- temperature ceramics (UHTCs) are better choices. Generally, these ceramic compounds are made of borides, carbides and nitrides such as ZrB2, HfB2, ZrC, HfC, TaC, HfN which are characterized by high melting points, high hardness, chemical inertness and relatively good resistance to oxidation in extreme environments, high thermal shock resistance [3,4]. But the only single-phase materials were not sufficient for high-temperature applications. Hence, many additives Nb, V,C, disilicate and silicon carbide were added to improve the resistance to oxidation in extreme environments. But UHTCs have high density compared to currently used materials. Hence in order to design TPS for maximum reuse and minimum weight, it is needed to go with some other UHTCs. Rapid advances in engineering design field lead to find out the alternate solution for conventional materials. Design engineers always looking for the material which gives better results than conventional materials in terms of weight, durability. From 1960 many of engineering applications in the world require high-temperature sustainability of materials. Most of the high-temperature field applications; SiC and Si3N4 are used as a primary high-temperature material. But this structural material does not withstand to recent temperature stability requirements. In recent scenario, a tremendous amount of interest is increased in an aerospace application, hypersonic concepts and weapons as well. To fulfil these high-temperature stability requirements there is need of new ultra-high temperature materials. Recent advance in TPS is needed for rockets, hypersonic flights, reentry vehicles to protect their orbiter from the extreme environment. While designing TPS orbiter temperature mustbe maintained at less than 177°C. To maintain orbiter temperature various materials are used TPS. In the modern era, space shuttle & hypersonic space vehicles require orbiter coatings that will ensure structures against temperature of more than 2000°C, enabling them to maintain compressive strength and high oxidation resistance. As of now utilized TPS materials give high strength and low density but having the low compressive strength and they oxidize in air at temperatures more than 500°C. Hence it is necessary to identify material which will overcome these shortfalls and will be the prominent solution for TPS. It has been decided to identify such material composition, synthesize and characterize them to test their suitability in TPS as a coatingmaterial.

The objectives for achievement of the aim of dissertation work are as follows-

1. To select a suitable composition to develop an ultra-high temperature ceramic composite for aerospaceapplications.
2. To carry out structural and thermal analysis of proposed composite in view of aerospace applications like wing of the space shuttle.
3. To synthesize ultra-high temperature ceramic composite and evaluate its properties.

To compare proposed composite with conventional materials and check its suitability for aerospace applications.

I. METHODOLOGY

The dissertation work is focused to carry out an analysis studyon a newly developed ultra-high temperature ceramic material.This material is exposed to various loads and temperatureconditions. The total proposed work divided in different phasesis as shown in fig. 2.1.

Methodology

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Material Selection for TPS

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Coupled thermal and structural analysis for proposed material by using suitable software

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Synthesize and experimental testing of ceramiccomposite

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Comparison of proposed material with conventional material

Fig. 2. Flowchart of the research plan

Phase I: Literature survey and selection of composition for proposed composite In this phase, the literature regarding ultra-high temperature ceramic composites, nano composites, oxidation and corrosion resistant materials, ceramic materials, processing and fabricationtechniques, experimental design and ANSYS software etc. will be studied.

Phase II: Structural analysis, Thermal analysis of the aerospace application (Wing) Preparation of wing model will be done according tospecifications. Loading conditions and boundary constraints willbe applied to the model by using suitable software. Structural and thermal analysis of composite wing will be carried out.

Phase III: Synthesis of ceramic composite material and evaluation of properties The Synthesis of composite according to the specificationsrequired for testing will be done by using powder metallurgy process. Various properties like density, young are modulus, tensile strength, etc. of composite will be evaluated experimentally.

Phase IV: Comparison and suitability Comparison of developed material with the conventional materials on the basis of mechanical properties will be done. Also, suitability of the developed material for aerospace application will be checked.

Closure this chapter gives information on various material used for thermal protection system (TPS). Also gives information about various ultra-high temperature ceramic composite, their properties and application in various fields.

2.1 Analysis of space shuttle wing

High-temperature reusable surface insulation tiles are used as TPS material for space shuttle wing. Here structure and thermal analysis are required for comparing properties like temperature distribution, heat flux transferred, and stresses developed in the wing for currently used material and selected composition of UHTCs.

2.1.1 Selection of material

The study is focused on the selection of suitable composition to define ultra-high temperature ceramic materials for space shuttle application. As the currently used materials have the low compressive strength and its oxidation start at 500°C, hence there is need to develop new material. The selection of new material is based on following criteria.

1. The requirement of the TPS of space shuttle wing by considering the flight parameters of the ATLLAS vehicle (speed,

altitude, etc.)

2. The shape of the wing (sweep back angle, tip radius, etc.)
3. The feasibility of processing method.

As per these criteria, selection of three compositions is done. These compositions are enlisted in table 2.1.1

Table 2.1.1: Composition of materials

Composition	Percentage Composition Details
Composition A	Al_2O_3 (48 %) + SiO_2 (43 %) + Fe_2O_3 (3.5 %) + TiO_2 (4.0 %) + CaO(0.5 %)
Composition B	Al_2O_3 (53 %) + SiO_2 (38 %) + Fe_2O_3 (2.5 %) + TiO_2 (3.0 %) + CaO (2.5 %)
Composition C	Al_2O_3 (58 %) + SiO_2 (32 %) + Fe_2O_3 (3.5 %) + TiO_2 (4.0 %) + CaO (1.5 %)

In this composition, silicon dioxide additive is used to decrease thermal conductivity and coefficient of thermal expansion of alumina. Titanium dioxide added to decrease sintering temperature and alumina densification. Calcium oxide additive is used to neutralize alumina.

2.1.2 Modeling of wing

Firstly, 3D model of a wing of the space shuttle is modeled byusing CATIA V5R120. Basically, two types of analysis are done on the model in Annoys Workbench 18.2. First, steady statethermal analysis is done by considering maximum temperature coming in the wing. Second, static structural analysis is done to understand Von misses' stresses coming in the wing as well as maximum deformation. As three compositions are selected forstudy, coupled thermal and structural analysis is carried out onabove selected compositions. On the basis of analysis result suitable composition is selected which is fabricated and experimental testing is done. Based on test results, suitability ofcomposition for respective space shuttle wing application will be tested.

Dimensions for Space Shuttle Wing:

- Overall length of the Shuttle= 23.42 m
- Width of the Shuttle = 9.05 m

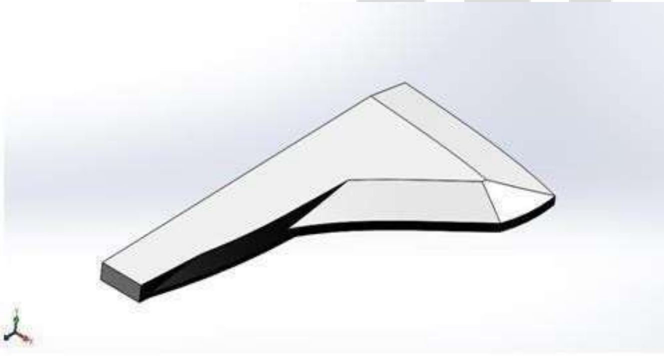


Fig. 2.1.2 CAD model of a Space Shuttle Wing

2.2 Coupled thermal and structural analysis

2.2.1. Material properties

Table 2.2.1: Material properties

Properties	Composition A	Composition B	Composition C
Density (Kg/m^3)	2306.66	2482.86	2514.9
Apparent porosity (%)	11.0	12.0	15.0
Cold crushing strength (MPa)	12.41	48.26	41.36
Thermal conductivity(W/m°K)	4.2	4.8	5.1
Thermal Expansion (10^{-6} /K)	1.4	1.8	2.1
Poisson's Ratio	0.21	0.21	0.22

Here ultra-high temperature ceramic for side wing body and lower wing body has been selected, the composition of the material is $Al_2O_3 + SiO_2 + Fe_2O_3 + TiO_2 + CaO$.

2.3. Thermal Analysis

A. Analysis result for current material (HRSI)

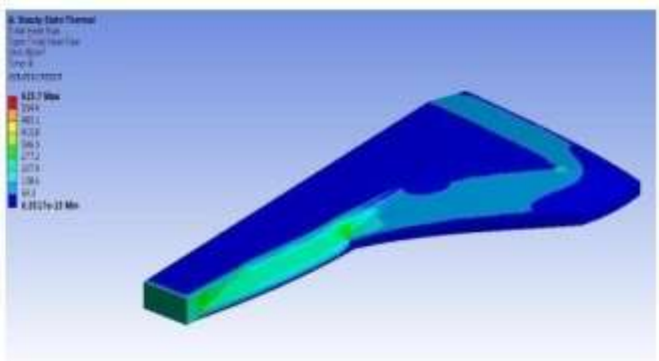


Fig.2.3. (a) temperate distribution in wing body forcurrent material

Fig. 2.3.(a) shows the maximum temperature of 1247°C, this is at the edges of the wing. The minimum temperature of 45.20°C, this is in the shuttle cabin. This is for a currently used material

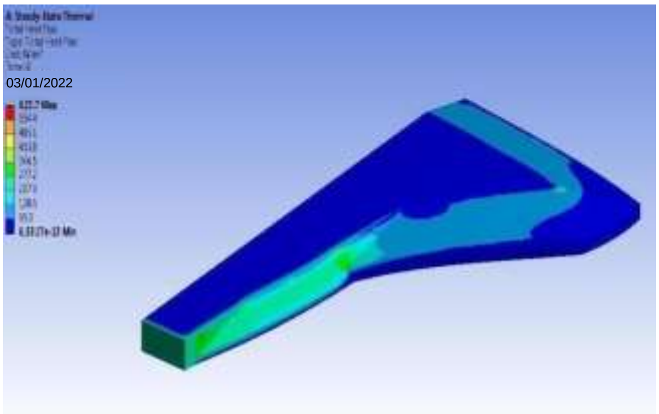


Fig.2.3. (b) Heat Flux in wing body for current material

Fig. 2.3.(b) shows maximum heat flux transferred in the wing bodyis 623.7 W/m^2. This is for a currently used material HRSItiles.

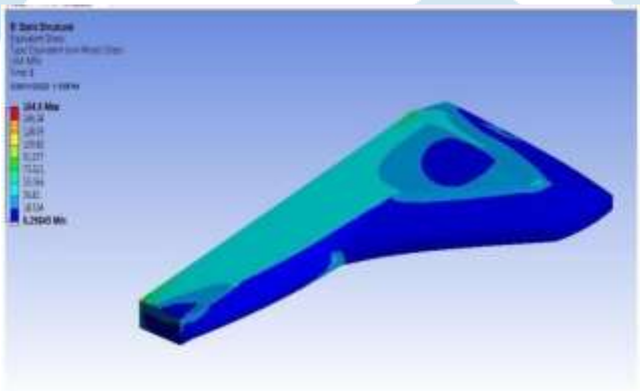


Fig. 2.3.(c)Stresses wing body for current material

Fig. 2.3.(c) shows maximum equivalent von-Mises Stresses developed in wing body is 164.6 MPa for a currently used material HRSI tiles.

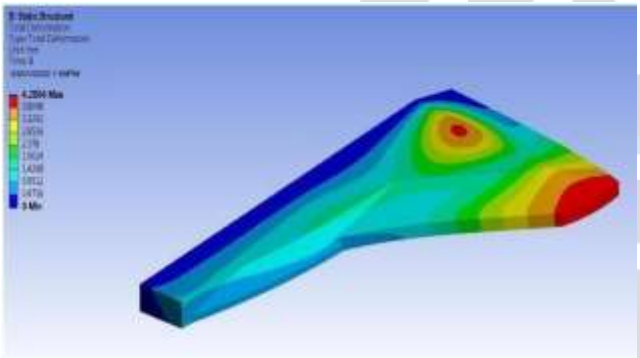


Fig. 2.3. (d) Deformation in wing body for current materialFig. 2.3.(d) shows maximum deformation in wing body is 4.28mm. This is for a currently used material HRSI tiles.
Steady state thermal analysis of the currently used material(HRSI) and three selected compositions gives temperature distribution and heat flux values in wing body. And static structural analysis gives equivalent von-Mises stresses and deformation in wing body. All these results are tabulated in following table

Table 2.3: Material Analysis for different Composition

Sr. No.	Material used for Analysis	Equivalent Von-Mises Stresses (MPa)	Deformation (mm)	Temperature (^oC)	Total Heat Flux (W/m^2)
1	Current Material (HRSI)	164.60	4.28	45.20	623.70
2	Composition A	166.09	4.46	51.29	735.15
3	Composition B	189.86	4.35	58.68	896.95
4	Composition C	250.7	4.98	71.41	946.27

Closure

In this chapter steady state thermal and structural analysis of currently used HRSI tiles and selected composition is done. In the numerical analysis, comparison of temperature distribution, heat flux transferred, developed von-Mises stresses and deformation in wing body is done. Finally based on numerical results composition A is selected for fabrication and testing.

2.4 Synthesis of Proposed Composite Material

Based on combined thermal and structural analysis of selected compositions it is observed that composition A that is Al_2O_3 (48 %) + SiO_2 (43 %) + Fe_2O_3 (3.5 %) + TiO_2 (4.0 %)

+ CaO (0.5 %) exhibits good results compared to composition Band composition C. Composition A is fabricated by hot pressing.

2.4.1. Fabrication methods

There are number of methods for manufacturing of ceramic composites. The main objective is to produce a composite of the desired shape with the desired microstructure. Ceramic composition, microstructure and properties are related as shown in fig. 2.4.1;

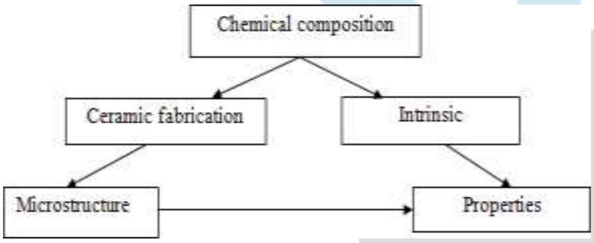


Fig.2.4.1 Relation between composition, microstructure and properties of ceramic [12]

The selection of fabrication method depends on whether the starting materials include a gaseous phase, a liquid phase, or a solid phase. As all materials are in powder form, the solid phase fabrication method is used. The manufacture from powder involves binding of particles of powders by the application of heat. Generally, two most common methods used for the manufacturing of ceramics. Melt casting. Firing of compacted powders (Sintering).

2.4.2 Raw material preparation

All ceramic powders are in a micron size with different mesh size. Hence it is needed to mix them properly. For proper mixing, ball mill is used. Ball mill consists of a hollow cylindrical shell rotating about its axis. It works on the principle of impact and attrition. The reduction in size is done by impact when the ball falls from the top of the shell. Stainless steel balls are used as grinding medium. All ceramic powders were mixed and blended in a horizontal ball mill as shown in the fig. 2.5.2 with following specifications

Density

The density of composite material depends upon the molecular interaction, the configuration of molecules, and porosity of the material. Specific gravity is calculated by using following equation;

$$\text{Specific gravity} = \frac{a}{a-b}$$

From the observations, $a = 11.04$ gms, and

$b = 5.55$ gms

$$= \frac{10.04}{10.04-5.55}$$

$$= 2.236$$

Now, $\text{Density} = 2.236. \times 1000$

$$= 2236\ \text{kg/m}^3$$

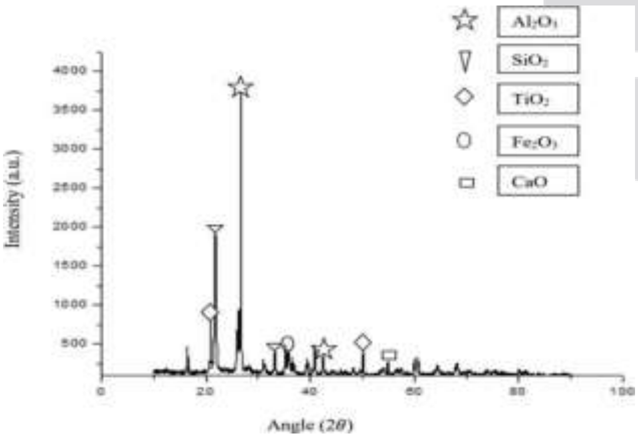
The density of 99.7 % pure Al2O3 is 3960 kg/m3 and that of SiO2 is 2650 kg/m3 while the density of proposed composite was found 2236 kg/m3. The HRSI tile which is base material hasa density equal to 2195 kg/m3 which was found near to the density of developed composite. The addition of low-density material like CaO, TiO2 results in the reduction of the overall density of the composite material. The porosity induced in the material during the hot pressing also reduces the density.

3.1 X-ray diffraction (XRD) test-

Closure

Fig. 2.4.2 Ball Mill

X-Ray Diffraction spectroscopy was utilized to verify the elements presents in composite phase. Fig. 6.1 shows the XRD pattern of the composite samples containing Al2O3, SiO2, TiO2, Fe2O3 and CaO particle reinforced ceramic matrix composite. The obtained pattern from the test is compared to standard XRD peaks of Al2O3, SiO2, TiO2, Fe2O3 and CaO available in the literature



The types of various solid-phase fabrication methods of ceramics are explained in this chapter. From these methods firing of compacted powder is selected for fabrication.
Firing of compacted method i.e., hot pressing method explained stepwise with required specifications.

II. PERFORMANCE EVALUATION

This chapter represents obtained from the test and experiment carried out in the previous chapter. Density, compressive strength, hardness and thermal stability of the currently used material and proposed composite are compared. X-ray diffraction pattern is plotted and identification of element is done. Fig. 3.2 XRD plot for Alumina Silica composite

From fig. 3.2 for Al_2O_3 particles peaks occur at an angle 26.68and 43 degrees. At 26.68 degree the reflection of X-ray light with a maximum intensity of 3727 units is observed. Similarly, for SiO_2 particles peaks occur at an angle 21.77 and 33 degrees. At 21.77 degree the reflection of X-ray light with a maximum intensity of 1929 units. For TiO_2 from above peaks occurs at an angle 21.69 and 50 degrees. At 21.69 degree the reflection of X-ray light with a maximum intensity of 842 units. For Fe_2O_3 peaks occur at an angle 35.26 degree and at this degreethe reflection of X-ray light with a maximum intensity of 490 units.

3.2 Rockwell hardness

Hardness greatly affects the resistance to wear, the strength of the composite. The value of hardness obtained from Rockwell hardness test for developed Alumina-Silica ceramic is 83.2 HRB. The hardness of developed composite was found greater than the base HRSI tiles. The incorporation of TiO_2 intothe material increases the overall hardness of composite. The particle size used for the material also governs the hardness of the material, smaller reinforcement results into higher hardness. The incorporation of TiO_2 and silica up to certain limit increase the hardness of material but after that, the material becomes brittle which is not desirable. Hence the silica is limited to 43%and TiO_2 is limited to only 4%.

3.4 Temperature stability

As the HRSI tiles are working in the temperature range of 649°C to 1260°C, the developed ceramic composite should sustain such high temperature. This property is very much important considering severity of the application as it is used for thermal protection system material. The developed composite was kept at a temperature of 1100°C for 900 seconds, it was reported that it can sustain such high temperature. The stability was confirmed by checking the initial and final dimensions of rectangular composite brick and strength of the same. As there was no change in the dimensions of brick and strength it can be said that the alumina ceramic composite can sustain temperature up to 1200°C.

Closure

The chapter presents the results obtained for density, compressive strength, temperature stability, hardness and XRD analysis of alumina-silica composite brick. The density of ceramic brick is nearly similar to currently used HRSI material. Compressive strength found greater than currently used material. The developed material was found thermally stable in working temperature range. In this dissertation work, Al_2O_3-SiO_2 was studied at 1247°C temperature and numerical analysis was carried outto finalize the composition among three different compositions. Simulated solutions at different nodes are validated with numerical solutions that are fairly in good understanding and show the feasibility of the problem methodology. From the numerical
For the composition ‘A’ the temperature distribution is from 1247°C to 51.29°C and the distribution is normal showing the uniformity of the distribution over the entire wing body.

1. The von-Mises stress developed in composition ‘A’ is 166.09MPa. Stress developed in composition B and composition C is 189.86 MPa and 250.7 MPa respectively, which are more than composition A. Thus, composition ‘A’ can sustain more thermal load than compositions ‘B’ and ‘C’.

2. Total heat flux in composition A is 735.15 W/m2. Total heat flux in composition B and composition C is 896.95 W/m2 and 946.27 W/m2 respectively, which is more than composition ‘A’?Thus composition ‘A’ has more resistance to transfer heat in wing body compare to compositions ‘B’ and ‘C’.

3. The composition ‘A’ gives better results compared to composition ‘B’ and composition ‘C’. Hence composition ‘A’ is finalized for synthesis.

4. While comparing composition ‘A’ with currently used material HRSI tiles there is not much difference between equivalent von-Mises stresses and total heat flux transferred.

The finalized composite was fabricated by hot pressing. The physical and mechanical properties of the developed composite were evaluated using experimental methods.

5. The density of alumina-silica ceramic composite is 1.86% more than the currently used HRSI material.
6. The compressive strength needed for thermal protection material to withstand against drag force. The compressive strength of alumina-silica brick (composition ‘A’) is 16.75 MPawhich is greater than currently used material HRSI tiles having 6.89Mpa
7. Alumina ceramic brick can easily sustain temperature up to 1200°C without losing its strength.

3.5 Future scope

The process followed and results obtained from the numerical and experimental investigation allow certain recommendations to be made that will be helpful in establishing future theoretical and experimental work. Dynamic numerical analysis can be performed to obtain more accurate results by considering close environmental conditions at high altitude and high temperature. At such severe conditions oxidation is a serious issue; hence oxidation test can be performed to check the material suitability more properly. The material at such conditions is subjected to continuous variation in the temperature, due to which it is necessary to check the performance of material with thermal shocks and thermal shock resistance property can be evaluated numerically and experimentally.

CONCLUSION

Hypersonic, re-entry vehicles or propulsion applications provide some unique thermal structural challenges (sharp leading edges, air intake). To meet the requirements of these components, certain specific materials are mandatory (UHTC). UHTCs are a promising technology used in many high- temperature structural applications. In this dissertation work, Al2O3-SiO2 was studied at 1247°C temperature and numerical analysis was carried out to finalize the composition among three different compositions. Simulated solutions at different nodes are validated with numerical solutions that are fairly in good understanding and show the feasibility of the problemmethodology

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