Overview of Nextel™ based structures for Space applications

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Abstract—Nextel Ceramic fibers are developed primarily for heat resistant applications. Due to their good strength to weight ratio, these fibers are finding extensive use both as reinforcement material and woven sheets used for various aerospace applications. This review deals with the role of Nextel founded structures for critical applications. These structures are employed as spacecraft shielding against debris attack and thermal insulation. Moreover the effect of different parameters including fiber/matrix interface and the microstructure for varying the properties of Nextel based structures are discussed.

Index Terms—Nextel fibers, Aerospace materials, interfacial coating on matrix, Thermal Blankets, Whipple Shield

1 Introduction

Space exploration has always been a challenging task for mankind and the quest for the best material suited for the specific applications is still attracting more and more researchers. In aerospace industry quality and durability is essential. Reason behind that is the outer space environment has requirement of such materials which possess excellent mechanical properties; coupled with high strength to weight ratio and resistance against different environments.

The research for best thermal insulation and the resistance against debris in lower earth orbit have lead to the invention of ceramic fibers which are mainly based on Alumina-silica compositions known as Nextel fibers. These fibers are processed as woven sheets, which exhibit much less weight than the green body made of identical composition.

Nextel based structures are being used in the aircraft engines, land-based turbines, rockets, hypersonic missiles, combustors, nozzles and thermal insulators. These fibers are mainly composed of alumina, but in their different grades, according to the requirement; silica, boria, zirconia, yttria, iron-oxide and mullite are added in compatible proportions to vary the properties of fiber. When these fibers are incorporated in ceramic matrix composites (CMC), their properties greatly depend upon the interaction and compatibility parameters between the fiber and matrix. These parameters can be changed by using different coatings on the fibers which play a major role in applications of CMCs. Coatings promote different mechanisms through which mechanical properties; primarily toughness and strength are altered.

This paper focuses on the Nextel founded structural systems that are presently being used for the specified applications of aerospace. These applications include chiefly the thermal insulation and the protection against debris impact.

2 High Performance Nextel Fibers

2.1 Composition

Oxide ceramic fibers, mainly based on alumina Al_2O_3 and Silica SiO_2 are favorable for refractory applications because of high stability of alumina at high temperatures. Minnesota Mining and Manufacturing (3M) Company is producing ceramic fibers with such composition. Commercially available fiber grades from 3M include Nextel 312, 440, 550, 610, 650 and 720. Typical properties of commercially available fibers are presented in Table I. Crystal size and filament diameter was reported as <500nm and 10-12µm respectively by 3M company. Nextel fibers preserve 75% of their properties above working temperature of 1000^{o}C [1].

2.2 Microstructure and mechanical performance

Nextel 312 and 440 are Alumino-boro-silicates, whose microstructure is composed of both crystalline and glassy phase. Glassy phase is due to the presence of boron oxide which has much lower melting point as compared to crystalline phases. Boron oxide acts as a glassy-viscous phase at the sintering temperature. Nextel grades namely 312, 440 and 550 enclose non-crystalline phases like silica and boron oxide which, due to their viscous behavior at high temperature, are unfavorable for creep performance [9].

Nextel 610, 650 and 720 have their crystal structure based on α- Al_2O_3 with absence of non-crystalline or glassy phase so they retain sufficient creep resistance and strength at higher temperatures. These fibers have good chemical stability and resistance to corrosion.

Pure alumina microstructure can only be found in Nextel 610. Apart from nearly 99% composition of Alumina, Nextel 610 contains approximately 0.67% Fe_2O_3 as nucleating agent and 0.35 % SiO_2 as grain growth inhibitor [1]. Nextel 610 has the highest tensile strength among the family of Nextel fibers but conversely its creep resistance is much less than the comparative grade (Nextel 720) which was specifically manufactured for its superior creep resistance [9].

Martin Schmucker and Peter Mechnich [10] found that the fine grain Nextel 610 fiber if heated to elevated temperature and embedded in porous alumina matrix, coarsening of grains occur. Figure 1 shows the SEM images of coarsening of grains after heat treatment at different temperatures.

TABLE I - PROPERTIES OF NEXTEL FIBERS

Property Profile	Unit	Nextel 312	Nextel 440	Nextel 550	Nextel 610	Nextel 650	Nextel 720
Chemical composition	wt%	62.5 Al_2O_3 24.5 SiO_2 13 B_2O_3[2]	70 Al_2O_3 28 SiO_2 2 B_2O_3[5]	73 Al_2O_3 27 SiO_2[8]	>99 Al_2O_3[2]	89 Al_2O_3 10 ZrO_2 1 Y_2O_3[2]	85 Al_2O_3 15 SiO_2[2]
Crystalline phase	-	Mullite+ Amorphous SiO_2[3]	γ -Al_2O_3+ Amorphous Mullite[6]	α -Al_2O_3+ Amorphous SiO_2[8]	α- Al_2O_3[2]	α- Al_2O_3+ cubic ZrO_2[2]	α- Al_2O_3+ Mullite[2]
Ultimate Tensile Strength*	GPa	1.7[3]	2.1[7]	2.0[15]	3.3[2]	2.5[2]	2.1[2]
Tensile modulus	GPa	150[3]	186[7]	193[15]	373[2]	358[2]	260[2]
Thermal Expansion	ppm/ ^{o}C	3[4]	5[7]	5[15]	7.9[2]	8.0[2]	6.0[2]
Density	g/cc	2.70[3]	3.05[7]	3.03[15]	3.90[2]	4.10[2]	3.40[2]

** 25.4 mm gauge*

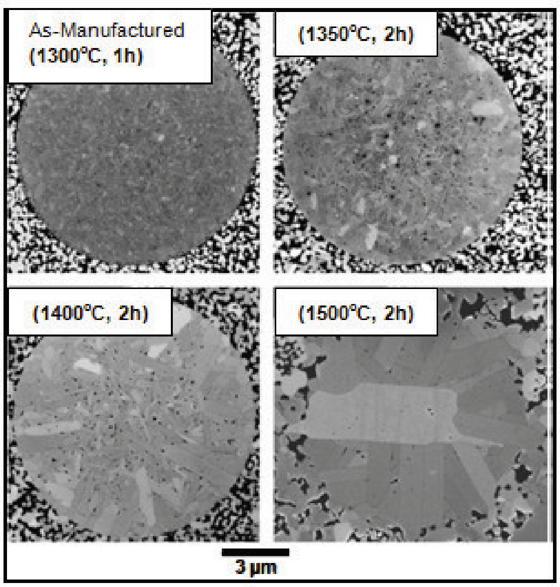

Figure 1 - SEM images showing coarsening of alumina grains after heat treatments [10].

Hence the microstructure of this fiber, embedded in porous alumina, is not stable at high temperature. Comparatively, if Nextel 610 is embedded in alumino-silicate matrix, then coarsening of grains can be suppressed at the price of strength [10]. Nextel 720 with 0.5µm globular mullite grains; five times than the size for Nextel 610[9] results in much better creep resistance than Nextel 610 but consequently its strength is low. Creep resistance is a result of reduced grain boundary sliding due to presence of acicular and globular grains [9]. The presence of mullite is approximately calculated as 55 vol. % and that of alumina is 45 vol. % [11]. Figure 2 shows a microstructure of Nextel 720 consisting of needle shaped mullite crystals neighboring alumina grains.

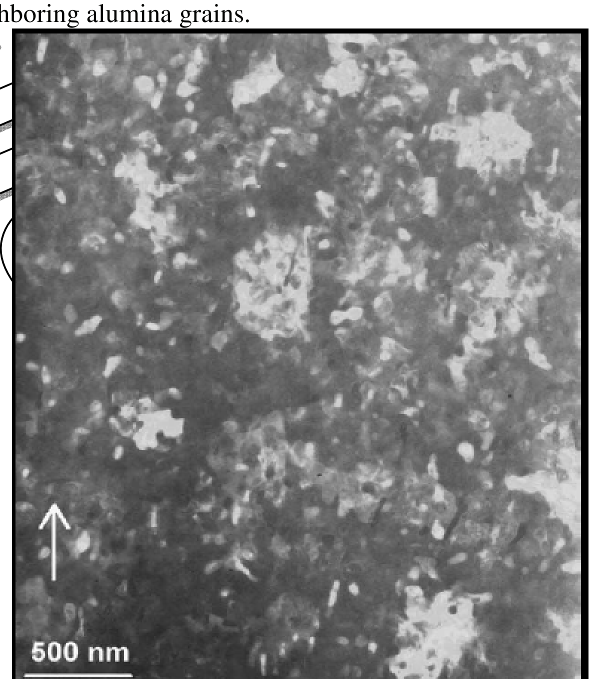

Figure 2 - Microstructure of Nextel 720[12].

Nextel 650 was manufactured to achieve better resistance against alkaline attack as compared to Nextel 720 and enhanced creep resistance as compared with Nextel 610. These improved properties are coupled with high strength[9]. The microstructure of the Nextel 650 is based on alumina grains with both inter and intra granular Zirconia grains [13]. Doping of Y_2O_3 is attributed to the reduction of creep in alumina due to the segregation of Y^{+3} ions at the grain boundaries hence limiting grain boundary diffusion [9].

3 FIBER/MATRIX INTERACTION

In the industrial application Nextel based ceramic matrix composites are being used widely in combustors, hypersonic missiles, thermal insulators, turbines and aircraft engines. The reason for their usage is their excellent strength, creep resistant behavior, lower densities and their good damage tolerance. As far as the mechanical properties are concerned the interfaces between fiber and matrix have great significance and much research studies have been made in this area.

In Nextel based composites a strong fiber/matrix interfacial bonding exists as a result these composites lack toughness and crack may propagate from matrix to fiber (see figure 3). To resolve this problem fibers are coated with various compounds. These compounds play their role in crack deflection and a potential improvement in fracture toughness just by introducing a separate interphase between fiber and matrix. The interface formed should be weak, otherwise crack will move throughout the structure resulting in low fracture toughness.

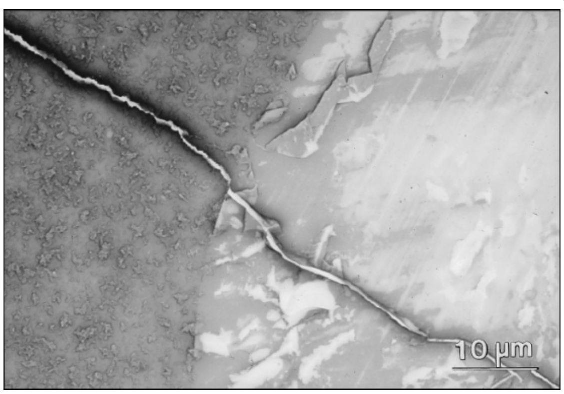

Figure 3 - Crack propagation into fiber [14].

L. R. Hwang et al. [3] noted that minimal interaction between the fiber and the matrix was essential for achieving good toughness in CMCs. For this purpose a thin barrier material is coated to prevent such interactions. S. Kooner et al. [15] reported that, when alumina fibers are coated with barium Zirconate and processed at high temperatures, reaction between fiber/coating and matrix/coating occurs and produces interphases, such as ZrO_2 and Ba/β-Al_2O_3, which are very promising for crack deflection and enhancement of toughness.

Interfaces [2] produce fibrous fracture in the composite material. In the case of Nextel 312 fiber, it is tested that Boron Nitride (BN) coating acts as a debonding interface layer. W. Zhao et al. [16] reported that this BN thin layer at the fiber/matrix interface is a very weak layer. It has two functions. One is to prevent the occurrence of chemical reactions and diffusion between the fibers and ceramic matrix and the second is toughening mechanism which is produced by debonding. Figure 4 shows a SEM image for the crack deflection around Nextel fiber coated with BN. Different processing routes and interphases are highlighted in table II.

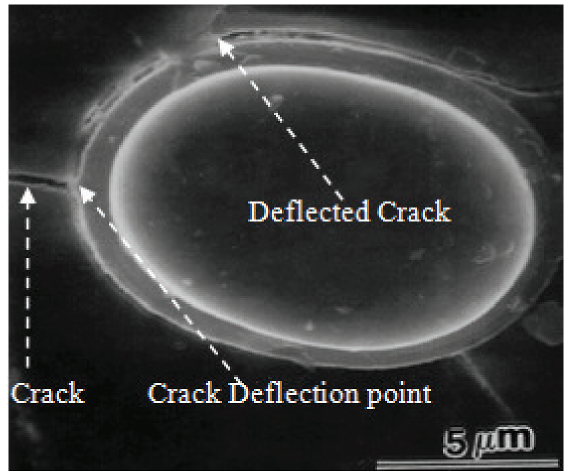

Figure 4 - Crack Deflection Mechanism in BN coated fibrous composites [17].

At high temperature these coating act as a barrier for fiber-fiber sintering hence eliminating weld-line defect. It also controls thermal etching which involves the surface diffusion of fiber atoms away from the grain boundaries [26]. These coatings also act as an obstruction for thermal grooving. When a crack is produced it propagates throughout the matrix, as it reaches the fiber its tip induces a shear stress on the fiber/matrix interface and causes decoupling [27]. The interfaces between fiber and matrix also enhance the damage tolerance. The phenomena occurs in such a way that weak fiber–matrix bond combined with the porous matrix produces a material structure that cannot develop sufficient strain energy to propagate a single dominate self-similar crack. Relative movement between the fiber and matrix enables crack deflection and increases toughness resulting in a CMC with damage tolerant behavior [28].

4 THERMAL INSULATION

Nextel fibers have recently found their major application in spacecrafts for thermal insulation. When space shuttle orbital returns back from space and enters in the earth's atmosphere, it experiences an enormous air friction. Due to this friction, temperature of the interacting material is increased. For this purpose a material is needed which can withstand high temperature.

Approximately 70% of the space shuttle must be protected against high temperature [29]. Ceramic fibers like Nextel 440, Nextel 312 and Nextel 550 are highly recommended fibers, which are either used with glass matrix or independently as fiber sheets for thermal protection and insulation. The primary factors for the usage of these fibers are their structure retention at high temperatures and light weight. J.B. Davis et al. [5] reported that Monazite-based coatings for thermal protection blankets are stable and compatible with Nextel 440 fibers at temperatures as high as 1200°C. These fibers are nowadays being used as a replacement for the silica-based compositions because of their much higher thermal protection. Figure 5 shows a thermal protection blanket used for aerospace applications.

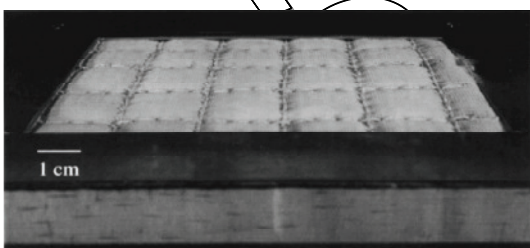

Figure 5 - Thermal protection blanket coupon for radiant heat and wind tunnel experiments [5].

Before designing an aerospace structure, the thermal shock resistance should be the core property of the material being used. Thermal shock is a phenomenon which is mostly experienced by space crafts, it is created by a sudden change in temperature and it can lead to fracture of the structure. Thermal shock involves both thermal expansion and thermal conductivity. Raj N. Singh and Hongyu Wang [30] reported that Nextel 312 embedded in SiC matrix showed significant retention of strength at different quenching temperatures difference (figure 6).

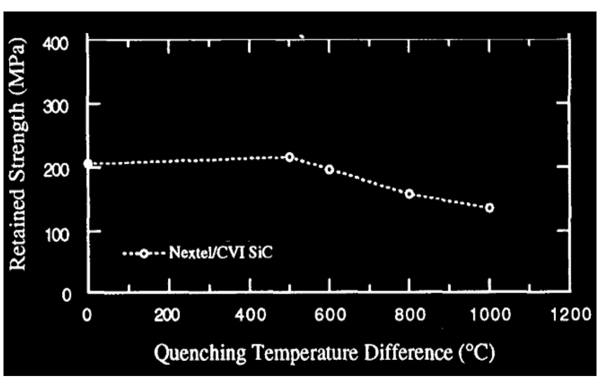

Figure 6 - Thermal shock behavior of Nextel/SiC composite [30].

Monolithic ceramics have also an ability to sustain a thermal shock but with the reinforcement of Nextel fibers it is increased. A comparative study of reinforced and unreinforced ceramic matrix showed that these fibers improved the thermal shock resistance of the matrix [31]. Thermal degradation also occurs in these fibers based composites and further studies confirmed that Nextel 720 with porous alumino-silicate matrix has showed promising results when tested at high temperature for a longer period of time [7].

5 SHIELDING AGAINST ORBITAL DEBRIS

A. Orbital debris

Orbital debris is ruins of the previous space missions which are orbiting the earth since their disintegration. According to a survey, the first orbital debris impact was confirmed by the NASA space mission STS-07. Space shuttle orbital vehicle (OV-099) showed a 3.8mm diameter and 0.43 mm deep pit on its right side middle window [32]. After impact on STS-07, huge research and development in the field of protection against the orbital debris have been carried out. The population of orbital debris is now increasing rapidly due to recurring space missions.

Size of orbital debris may be classified as large (Diameter>10cm), medium (Diameter 10cm-1mm) or small (Diameter<1mm) [33]. The population of orbital debris is summarized in table III. According to the data presented, small debris due to their size and population are potential threats for the space craft shields.

TABLE 3 - POPULATION OF ORBITAL DEBRIS

Size	Population	Percentage	Reference
Large	> 9 000	<0.1%	[34]
Medium	> 100,000	<1%	[34]
Small	>35,000,000	>99%	[34]

B. Hyper Velocity impact

Protection against space debris can be considered analogous to the ballistic protection for military mobile structures like vehicles, ships and aircrafts. The difference arises in the impacting velocity of space debris and ballistic impact. Velocity of space debris in lower earth orbit may vary from 1000m/s to 15000m/s [35] which has a significant effect on the impact bearing capacity of the material shield used. Due to hyper velocity impact, a shock wave is produced in material, which causes extensive amount of energy generation, which may melt the debris, evaporate or change its phase depending upon the velocity of the impact. The impact to the material causes both the material and the debris to form fragments. These fragments referred to as "debris cloud" will now penetrate inside the shielding and would interact with the inner walls. Keeping this in view, inner walls of the shield are made in such a way that they minimizes the effect and hence provide minimum or least damage to the space craft

TABLE 2 - PROCESSING ROUTES OF NEXTEL BASED CMC'S AND THEIR STUDIED PROPERTIES

Nextel Fiber	Matrix	Interphase	Process	Researched Properties	Reference
610	High purity alumina powder	Monazite	Slurry infiltration	230 MPa (UTS)	[18]
480	Mullite powder	BN	Slurry infiltration	258 MPa (Max. Stress)	[8]
550	Mullite powder	BN + SiC	Slurry infiltration	223 MPa (Max. Stress)	[8]
720	Alumina	Porous alumina	Sol-gel	55 MPa (UTS)	[19]
720	Mullite	Porous Mullite	Slurry infiltration	160 MPa (Bending Strength)	[20]
720	Alpha-Alumina	$NdPO_4$	Electrophoretic Deposition	142 MPa (Tensile strength at room temperature)	[21]
720	Alpha-Alumina	ZrO_2	Electrophoretic Deposition	136 MPa (Tensile strength at room temperature)	[21]
610	Mullite	Carbon	Polymer Pyrolysis	177.4 MPa (Tensile Strength)	[22]
610	Porous Alumina	Monazite	Filament winding	37% improvement in UTS	[23]
312	Blackglass(Si-C-O)	Carbon	Polymer pyrolysis	240 MPa (Flexural Strength)	[3]
720	Reaction Bonded mullite	$AlPO_4$	Electrophoretic Deposition	175 MPa (Ultimate Bend Strength)	[24]
720	Alumina-silica	No coating	Electrophoretic Deposition	1500 MPa (Tensile Strength at 1200^0C)	[25]

C. Whipple shield

Millimeter or sub-millimeter space debris having a hyper velocity has a tendency to perforate the external body of the space craft. Ordinary material cannot become impediment in the way of incoming debris. So, a multi layer sheet known as Whipple shield is used for space craft shielding against debris impact.

Whipple shield consists of a front Al-Alloy bumper and an intermediate layer or layers and then a rear wall. All the layers have spacing between them so as to allow the fragments to decrease their impacting load. It is found that [36] All-Aluminum Whipple shields consisting of Al-Alloys front bumper and rear wall with no intermediate layer were not much effective in decreasing the impact velocity and it also caused much damage to the rear wall. Moreover the use of thicker bumper caused excessive load hence it is not much weight effective. As an option, use of Nextel/Kevlar based stuffed intermediate layer offer many advantages. The Nextel/Kevlar intermediate shield, the aluminum outer bumper and rear walls is known as Stuffed Whipple shield. It is reported [36] that Stuffed Whipple shields were successful in stopping 50-300% more massive projectiles compared to Double bumper Aluminum shields of equal length. Also Nextel is better in bearing the impact of projectile fragments than Aluminum.

Stuffed Whipple shield consists of intermediate plate which is composed of mostly used Nextel grade i.e. Nextel 312 with designation of AF-62 along with Epoxy resin based Kevlar plies. Mostly two to six layers of both Nextel and Kevlar are used for formation of intermediate layer [36]. Figure 7 shows the schematic of the Stuffed Whipple shield while Figure 8 shows the schematic of impact of debris and debris cloud. Experimental results show that the stuffed Whipple shield allows following advantages [37] over All-Aluminum Non-Stuffed Whipple shield:

- Efficient conversion of projectile kinetic energy into thermal energy.
- Produces less damaging debris cloud.
- Less sensitivity to projectile shape.

- Less cumulative damage to rear bumper or pressure wall.
- Weight and space saving benefit

Hence it is believed that, stuffed Whipple shield is the best obstacle for the projectiles.

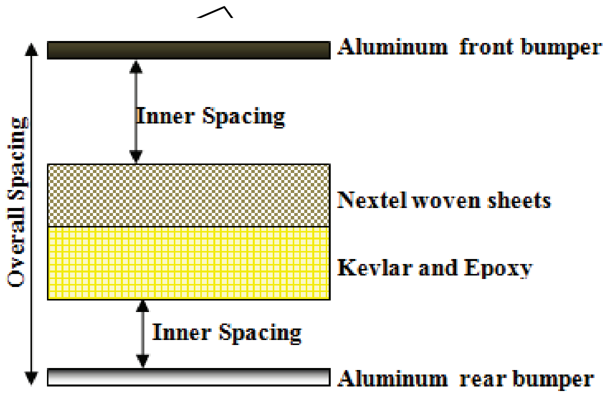

Figure 7 - Schematics of stuffed Whipple shield

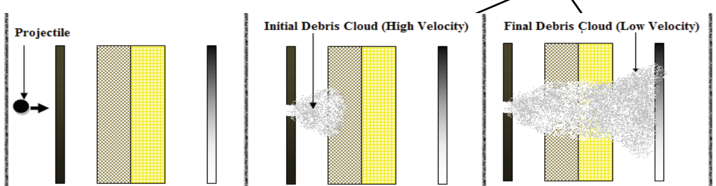

Figure 8 - Schematic of impact of debris and debris cloud.

6 CONCLUSION

Nextel based structures are finding rapid importance in the structural applications for spacecrafts. Several grades of 3M Nextel fibers are overviewed according to mechanical properties like Nextel 610 is better than 720 in an application where higher strength is required, whereas Nextel 720 has high creep resistance. Nextel fibers including 440, 312 and 550 are recommended for the applications requiring thermal insulation. So these fibers are very high performance reinforcement materials for the CMCs, being used in aerospace structures. The debris effect is studied along with the Whipple Shield protection, which is used to protect the outer body of the space craft. Nextel fibers based composite blankets are believed to be nearly ideal structure for thermal insulation of spacecrafts but there is scope of further research. Matrix/Fiber interface and crack propagation phenomena are revised and it is an open research area.

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