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Integrity of Structural and Thermo-Structural Materials for Indian Space Programme

A.K. Shukla, V.M.J. Sharma, S.V.S. Narayana Murty, P. Ramesh Narayanan and S.C. Sharma*

Materials and Metallurgy Group, Vikram Sarabhai Space Centre, Trivandrum-695 022, India.
*E-mail ID: sharma_sc@vssc.gov.in

Abstract

As stakes are very high, a space programme places one of the highest priorities on the integrity of structural materials and Indian space programme is no exception to this. Starting from design of process, an exhaustive series of structural simulations, mechanical and structural tests and inspection-steps at room, elevated and cryogenic temperatures are followed during qualification and acceptance of structural materials for space applications. A continuous effort of space material-scientists is to adopt the emerging advanced destructive and non-destructive testing methods in order to enhance the reliability of structures. In the second phase of Indian space programme, high temperature materials are poised to play increasingly more important role. This in turn, calls upon the scientists to develop a variety of thermo-structural materials based on dispersion strengthened alloys, ceramic matrix composites, intermetallics, ultra high temperature ceramics etc. Keeping pace with these developments, a host of characterization techniques are being evolved and facilities are being established to ensure the thermo-structural integrity of high temperature materials. The paper presents the whole gamut of important structural and thermo-structural materials that are presently in use or under development for the future missions of ISRO. It also includes the testing methodologies being adopted at VSSC/ISRO for qualifying these materials from the structural integrity point of view.

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1. Introduction

Recent technological advancements in the aerospace, nuclear, chemical and the energy sector are presenting the challenge of developing advanced materials which can perform under more hostile operating conditions. The materials and structures are expected to experience higher operating stress levels, application temperatures and aggressive environments [1]. The constant effort of the material scientist is to understand the application requirements and subsequently, to strive for developing a new material to suit the application.
requirements or to improve the performance of an existing material by modifying the processing-microstructure related aspects.

Structural integrity is concerned with determining and predicting the performance, failure, durability and safety of the fabricated component that is subjected to a range of operating conditions during usage. The principal disciplines involved in predictive structural integrity are materials modeling, stress analysis, inspection techniques and experimental validation. The damage caused due to faulty heat treatment, welding, material degradation will result in different types of failures depending on component size and loading conditions. Hence, it is not sufficient to only understand the failure mechanism, but establishment of microstructure-property correlation is also essential. Therefore research needs to focus and establish correlations between materials development, laboratory testing, numerical modeling, operating conditions and failure modes. This will permit a faster development cycle for improved and optimized material production leading to excellent integrity of components even under extreme service conditions.

2. Structural and Thermo-Structural Integrity of Aerospace Structures

2(a). Structural Integrity-An Example of Solid Rocket Motor Casings

Solid rocket motors are the main source of power for rockets. Many of the operational satellite launch vehicles and missiles around the world depend on solid motors for their propulsion during the initial phase of flight. In solid motors, propellant housed inside the combustion chamber is burned in a controlled way to get the desired thrust profile [2]. Solid propellant burning can be considered as controlled explosion. To ensure control over the burning, the propellant grain and interfaces should be free from critical defects. With the advanced analytical and manufacturing capability solid motors are designed and realized without major flaws. Large solid boosters are processed in segments and assembled together (Fig. 1). ISRO has qualified solid motors with 200 tonnes of propellant loading [3]. Making solid propellant grain is an art more than engineering. The solid ingredients are mechanically mixed along with binder and chemically cured. Due to various reasons, presence of certain defects in the composite propellant grain and interfaces cannot be ruled out. For the acceptance of propellant system for the specific application, soundness of the grain and integrity of interfaces are very important in addition to achieving the specified mechanical and interfaces properties. Unlike liquid engines solid motors do not get tested before their use. This highlights the importance of non-destructive evaluation (NDE). Normal functioning of a solid motor can be ensured, if the subsystems and interfaces are free from critical defects [4].

Another important aspect of the solid rocket motors is the integrity of the pressure vessel. Pressure vessels generally contain crack like defects which are either inherent in the material or get introduced during the fabrication process. These cracks usually have sharp edges and are sensitive to crack growth and fracture. The increased use of modern high strength materials while producing lighter products, tend to be less forgiving for the presence of defects than the traditional ductile materials. A pressure vessel made of these materials is likely to have issues with respect to structural integrity in the presence of flaw/crack size not detectable by conventional NDE methods.

The types of flaws/defects that are usually encountered in metallic pressure vessels are: (i) visual surface defects such as cracks, pitting, corrosion defects and dents; (ii) local thickness reduction due to erosion and manufacturing process; (iii) defects such as porosity, undercuts, slag inclusion, cracks, piping in the welded regions and some of these defects may even exist in the manufacturing process of the sheet material; and (iv) defects while machining, deep tool marks and poor surface finish. These defects are categorized as non-planar or volumetric defects (e.g. porosity, slag inclusions) and planar defects (e.g. cracks, lack of side wall fusion). When the defects in a pressure vessel are detected through NDE techniques, the decision to use/reject the component with/without repair is a challenging task. The decision will involve such problems as safety of the personnel, safety of pressure vessel, repair cost and time delays in the project execution. For large pressure vessels, repair costs are high, but on the other hand possible damage due to a failure of a pressure vessel could be enormous and should be avoided. Consequently, a sound engineering judgement on the safety of large pressure vessels which contain defects is extremely important. In spite of the best engineering practices and manufacturing techniques, it is practically impossible to realise a defect free pressure vessel. Therefore, it is important to understand the repercussions of the presence of defects. This demands complete understanding of the material behaviour, operating conditions, reliability of NDE techniques to arrive at a decision on the flightworthiness of the structure.
Table-1 presents the summary of materials and their properties usually used for rocket motor application [5]. Since these are high strength materials, resistance of which is sensitive to presence of crack like defects, it is required to establish fully controlled design and quality control procedures to realise a reliable hardware with the desired design margins.

Table-1: Summary of materials and typical properties for solid rocket motor applications.

| Properties       | 300M | 4340 | 15CDV6 | 0.3C-Mod. CDV6 | D6AC | 18Ni (250) Maraging steel | CF250 Maraging steel |
|------------------|------|------|--------|----------------|------|--------------------------|---------------------|
| Hardness (HRC)   | 52   | -    | 35     | 42             | -    | 52                       | 51                  |
| 0.2%PS (MPa)     | 1550 | 1080 | 850    | 1300           | 1520 | 1725                     | 1675                |
| UTS (MPa)        | 2100 | 1420 | 1000   | 1400           | 1790 | 1780                     | 1750                |
| % Elongation     | 8    | 10   | 12     | 8              | 5    | 10                       | 10                  |
| % RA             | 30   | 15   | -      | -              | 18   | 50                       | 50                  |
| $K_{IC}$ (MPa√m) | 71   | 71   | -      | 80             | 80   | 90                       | 95                  |
| $K_{Ic}$ (MPa√m) | 19   | 16   | -      | -              | -    | 33                       |                     |
| $E$ (GPa)        | 193  | 200  | 210    | 210            | -    | 180                      | 180                 |
| Impact at RT (J) | 25   | -    | 25     | 15             | -    | 18                       | 20                  |

2(b). Thermo-Structural Integrity-An Example of a Regeneratively Cooled Thrust Chamber

The design of hot/thermal structures for high performance flight vehicles has placed ever increasing demands on material performance. Structural designs for aerospace use typically require high-stiffness, thin-gauge materials that can be fabricated into complex structures. Thermo-structural designs typically require high strength, low density materials that retain their desirable properties even at high temperatures. Moreover it is known that higher operating temperature result in higher operational efficiency [6]. Further, materials for thermo-structural applications should retain their microstructure as well as mechanical properties even after prolonged exposure to
elevated temperatures. Therefore, there is a substantial research effort currently underway to develop new aerospace materials to meet these challenges.

The potential materials to meet the challenges of high performance flight vehicles are intermetallics (particularly aluminides of nickel and titanium), metal matrix composites (ceramic dispersed metal composites), ceramic matrix composites (C-SiC, SiC-SiC, ZrB2-SiC) and carbon-carbon composites. Intermetallic materials particularly nickel and titanium aluminides are promising materials for replacing the super alloys [7]. Even though these intermetallics possess superior specific strength, their room temperature ductility is unacceptably low for processing into usable product shapes. Therefore, efforts are on to improve the room temperature ductility of these materials. Hence, both material and process improvements have to be made before the promise can turn into performance of these materials.

One of the important and structurally critical elements in a rocket engine is the thrust chamber where ignition of the propellants takes place. In a cryogenic engine operating on liquid hydrogen and liquid oxygen system, a regeneratively cooled thrust chamber is usually used. A regeneratively cooled thrust chamber consists of channels for the passage of liquid hydrogen made by brazing of an inner shell with milled channels to that of a steel outer shell. In view of the high thermal conductivity, copper alloys are used for the thrust chamber inner wall which is in contact with the hot gases. The thermo-structural integrity of the thrust chamber has to be ensured since the hot wall side is exposed to high temperature and hydrogen admitted at a lower temperature is passed through the channels. The brazed joint is vital to the performance of the thrust chamber and should retain the structural integrity throughout the flight. Figure 2 shows the schematic of a regeneratively cooled thrust chamber showing different elements along with the photograph of a common abnormality noticed in the system showing the yielding of the channels showing ‘dog house’ effect. This is further complicated in a semi-cryogenic engine employing kerosene and liquid oxygen system where the channels may get blocked due to ‘coking’ leading to an increase in the local temperature. This can be rectified by the selection of proper grade fuel with controlled impurity levels.

Figure 2: Schematic of a regeneratively cooled rocket thrust chamber and (right) schematic showing ‘dog house effect’ [1].

Copper based materials are considered to be candidate for high temperature applications in rocket engines, which warrant high strength, high thermal conductivity, resistance to creep and low cycle fatigue. Applications such as combustion chambers, nozzle liners, high performance metal gaskets and various other technologies of expendable rockets as well as RLVs [1] also require Cu-based materials with the above mentioned properties. A schematic representation of a combustion chamber used in rocket engines is shown in Fig. 2. Various components such as combustion chamber liner, injector face plate and protruding type injector elements demand Cu-based materials with enhanced thermal/mechanical properties. These components experience very high temperature, which is the result of combustion of gases (≈2700 °C) and hence, are prevented from melting through cooling by cryogenic fluids (liquid hydrogen) flowing through them [1].
One of the critical facilities required for establishment of design data for the brazed joints of copper to steel used in regeneratively cooled nozzles is a vacuum brazing furnace with controlled cooling and heating facilities. This is in view of the fact that the braze foils are highly reactive nature and their surface contamination can lead to poor joint quality. Further, controlled heating and cooling are essential to achieve uniform temperature in the substrate and braze foils as well as to minimise the evaporation losses/thermal stresses. Figure 3 shows a vacuum brazing furnace with gas quenching facility. A typical photograph of a helical channel milled copper divergent nozzle of a regeneratively cooled rocket thrust chamber is also shown in the Fig. 3. Further, a new generation copper alloy Cu-8Cr-4Nb processed through vacuum hot pressing [8] using in-house facilities at VSSC is also shown in the Fig. 3. Typical values of different properties for various copper based alloys and compounds are also presented in Table-2.

Figure 3: (left) Vacuum brazing furnace with gas quenching facility; (middle) a regeneratively cooled thrust chamber liner with helical nozzles made of copper alloy; and (right) next generation Cu-8Cr-4Nb alloy for thrust chamber liners processed by vacuum hot pressing.

Table-2 Summary of the materials for regeneratively cooled rocket thrust chambers (SS-solid solution treated) [9].

| Material          | Hardening phase | Processing Method       | Microstructural Stability (above 500 °C) | UTS (MPa) at RT | Thermal Conductivity at RT (W/m-K) | Oxidation Resistance |
|-------------------|-----------------|-------------------------|------------------------------------------|----------------|-----------------------------------|---------------------|
| OFHC Cu           | -               | Casting                 | Poor                                     | 200            | 397                               | Poor                |
| Cu-Cr             | Elemental Cr    | Casting + SS + Aging    | Poor                                     | 500            | 300                               | Poor                |
| Cu-Cr-Zr-Ti       | Intermetallic phases of Zr, Ti | Casting + SS + Aging | Poor                                     | 550            | 250                               | Poor                |
| Cu-Zr             | Intermetallic phases of Zr with Cu | Casting + SS + Aging | Poor                                     | 450            | 350                               | Poor                |
| Cu-Ag-Zr          | Elemental Ag, intermetallic phases of Zr | Casting + SS + Aging | Poor                                     | 350            | 300                               | Poor                |
| Cu-Al₂O₃          | Al₂O₃           | Atomization + Internal Oxidation + Consolidation | Excellent | 400            | 340                               | Good                |
| Cu-Cr-Nb          | Cr₂Nb           | Atomization + Consolidation | Excellent | 400            | 300                               | Excellent           |

Thermo-structural materials in re-entry/re-usable vehicles also include thermal protection materials. Thermal protection systems (TPS) are essential to protect the structural integrity of re-entry/re-usable vehicles during atmospheric re-entry which causes severe aerodynamic heating [10]. The TPS approach has the advantage that insulating materials are considered separately from the structural elements. Insulation can be designed to withstand...
predicted heating conditions whereas the structural elements can be designed to withstand structural loads such as vibration, acceleration, thermal shock and particle erosion independently. The insulation and structural elements are then integrated to form the structure of the vehicle.

The philosophy of thermal protection systems is two pronged. First is to use the thermo-structural materials which can withstand the re-entry environments, which are often called hot structures. Second approach is to use coatings to thermally protect the inner cold/vulnerable structures. The hot structure philosophy is applied to areas expecting excessive thermal loads or where the structural thickness does not allow integration of an additional TPS. These components include nose cone, leading edges and engine air intake elements. In a cold structure philosophy, the structure is cooled by active/passive cooling aided by thermal barrier coatings.

2(c) Operating Environments

Operating environments in rocketry vary from extremely low temperatures (viz. 20K for liquid hydrogen and 80K for liquid oxygen which are the propellants in a cryogenic engine) to extremely high temperatures (viz. those experienced in the thrust chambers). The conditions of static/dynamic loads also vary significantly depending of the launch phase. The materials during the processing, fabrication and usage are subjected to different types of environments (viz. marine environment as most of the launch sites are near to sea coast or in some cases corrosive propellants like Hydrazine, N₂O₄ or RFNA) storage conditions. Depending of the material under consideration and severity of environment, the materials have to be screened for corrosion and stress corrosion susceptibility. Similarly, thermostructural materials face oxidizing environments and hence evaluation of oxidation resistance is also essential. Figure 4 shows the facilities for testing from 4K to high temperatures along with environmental susceptibility testing.

![Figure 4](image_url)

Figure 4: State of the art test machines (a) for testing from 4K to room temperature; (b) high temperature test facility and (c) slow strain rate test machine for studying the environmental susceptibility.

3. Evaluation Methods For Structural and Thermo-Structural Materials

High standard of reliability requirements of launch vehicle structures calls for a number of tests and simulations. The test programme is intended and designed to ensure the satisfactory performance of the launch vehicle structures with adequate margin to compensate/nullify the fluctuations during actual use of the system. These tests are generally classified as follows:
a) Material characterization which is meant to generate input data to design various elements of the launch vehicle structures viz., load bearing members, thermal protection system, interface integrity. These tests are performed under flight typical/specified environment and to determine erosion/oxidation of the material.

b) Part component level testing such as proof testing of fasteners. These are intended to qualify individual structural element system. These tests are carried out for evaluation of behaviour under simulated mechanical, aero-thermal environments.

c) Life estimation tests/ demonstration of adequate performance at the intended life period. The structural elements including those load bearing members are subjected to complete load cycle of actual/simulated environment. These tests are intended to be carried out up to failure of the system component to estimate its safe life operation.

d) Prototype testing: Full structural system is tested to demonstrate its performance under simulated environment. The working life of the system shall be translated into initial design margins and tests are conducted to demonstrate these margins. Some of specifics of space qualification are the testing of components under:
   - Vacuum conditions, to test for degradation and outgassing (test in a decompression chamber)
   - Space/solar radiation, to test for degradation and thermo-optical characteristics (test in an environmental chamber); particle bombardment, to test for resistance against rain, ice, small meteorites and space debris;
   - Low/high-frequency oscillations, to test for resistance against launch vibrations and random noise.

e) High temperature testing facilities for hot structures and structures with large thermal gradients are required for the qualification and evaluation of systems that have structures exposed to very high temperature. To simulate the large thermal gradients, a high temperature kinetic heating simulator facility is a necessity.

3(a). Material Property Evaluation

The characterization of mechanical behaviour of materials is very important for the development of newer materials and processing techniques. The test data is essential for designers for design optimization of various components. The high temperature material characterization is extremely difficult for a plethora of reasons. Firstly, the data is often required up to very high temperatures – sometimes very close to the melting temperature. At such high temperatures, the design of test accessories like heating source, grips and extensometers becomes challenging. Repeat/confirmatory tests are often required to establish the lower and upper bounds of the data range as well as to ensure that the data is of adequate accuracy. The high temperature materials themselves are very expensive and only limited quantities/sizes and shapes are available especially during prototyping of new grades of alloys. All these factors make high temperature testing costly and time consuming. Secondly, the hot structural materials are often used in thin sections (due to weight reduction criteria) which necessitate testing of thin sections adding to the existing problems. Lastly, the service conditions are not precisely defined due to uncertainties involved in assessment of service conditions.

Materials exposed to high temperatures such as turbine hot section materials are subjected to variable structural loads (pressure and centrifugal) as well as variable thermal loads due to start ups and shut-downs which results in thermomechanical fatigue (TMF) condition. Under these conditions three kinds of degradations lead the material to failure: creep, fatigue and oxidation. The individual extent of these damage mechanisms and their mutual and synergistic interactions depend very strongly on the specific material under study and the loading conditions imparted. Development of an accurate concept to predict life expectancy of metallic materials subjected to complex loading conditions is a prerequisite for improving reliability and economy of the use of such materials. Generating precise thermo-mechanical deformation data is therefore essential to support constitutive model development for accurate life time estimation. Here, the requirement is for experimental data that is free from errors caused by less than ideal equipment and procedures. Specifically, problems that are generally encountered in TMF testing (Fig. 5) are related to specimen stability, thermal strain compensation and temperature/mechanical strain phasing.
A.K. Shukla et al. / Procedia Engineering 86 (2014) 8 – 17

Figure 5: Thermomechanical fatigue test under progress

3(b). Special Test Facilities for High Temperature/Thermal Protection Materials

Development and qualification of thermostructural materials/thermal protection systems for rockets and re-entry vehicles requires extensive laboratory level testing to study material performance. High enthalpy facilities have proved to be one of the most useful laboratory tools for thermal testing at high temperatures/heat flux for the intended test durations simulating those encountered during flight conditions. The technology being classified, establishment of such advanced facilities was essential and thus initiated in VSSC (Fig. 6) [11]. These facilities are being used regularly to evaluate the performance of different types of thermo-structural/thermal protection materials being developed for rockets and re-entry vehicles.

Various tests carried out in these facilities include material characterization tests, design validation tests and certification tests. Typical materials which are tested include ablative materials like Carbon-Carbon, Carbon phenolic, silica phenolic, graphite etc., high temperature metallic materials such as silicide coated columbium nozzles, ceramic TPS materials such as silica tiles, Ceramic Matrix Composites, Zirconium diboride etc. Parameters simulated in arc plasma tests for evaluating TPS material performance are heat flux, enthalpy and test duration. Heat flux from 50 W/cm² to 1500 W/cm² and temperature up to 6000 K are simulated in atmospheric arc plasma generator facility. 1 MW High enthalpy facility and 6 MW plasma wind tunnel facilities are the unique facilities for re-entry simulation testing. These facilities play a crucial role for qualification of TPS materials under extreme thermal environments prevailing during re-entry. Enthalpies up to 35 MJ/Kg, temperatures up to 9000 K along with prevailing heat flux, ambient pressure and the hypersonic flow over the test model are also simulated in these facilities. Data generated on material performance consists of surface and sub-surface temperature history, heat of ablation, erosion rate, mass loss rate and material behaviour with surface chemical reactions. Specific tests to study the effectiveness of oxidation resistant coatings and impact analysis can also be carried out.

Figure 6: Testing under progress in (a) Plasma jet facility and (b) high enthalpy facility.
4. Failure of Materials Used in Aerospace Structures

Stronger and lighter materials are the norm during the selection of materials for aerospace applications in view of the inherent advantages of carrying higher payload for a given mass of structure. However, high strength materials are also prone for environmental assisted cracking [12]. High strength is derived from the metastable nature of the microstructure (viz. peak aging to attain highest strength) and specific tempers are developed (for example T73 temper for aluminium alloy AA7075) for materials to avoid the problem of stress corrosion cracking. Therefore, caution must be exercised while selecting the material and high strength alloys should be considered, only in cases, where there is no obvious choice. Further, preference should be given for temper conditions which have demonstrated resistance for stress corrosion cracking. Since stress corrosion cracking occurs when a combination of tensile stress, a corrosive environment and susceptible microstructure are present together, attempts should be made to reduce the tensile stress (inclusive of assembly and residual stresses) as well as protect the structure from exposure to the environment through suggested protection methods. Figure- shows the failure of structural components viz. stress corrosion cracking in M250 maraging steel observed as intergranular cracks in an optical microscope and hydrogen embrittlement in 35NCD16 steel observed as brittle faceted fracture observed in a scanning electron microscope (Fig. 7).

Figure 7: Stress corrosion cracks in a M250 grade maraging steel and (b) hydrogen embrittlement in a 35NCD16 steel.

5. Summary

The structural and thermo-structural integrity aspects of materials used in launch vehicles are presented with specific examples from solid rocket booster casings and regeneratively cooled thrust chambers. Further, the intricacies of testing and qualifying structural and thermo-structural materials for aerospace applications are also presented.

In spite of the best efforts right from materials selection, processing, fabrication and storage, failures of structural members are not uncommon. While the selection of material is based on the test data for a particular application, the processing, characterization and qualification of materials for aerospace applications is quite a challenging task. Once the material is certified for usage, it has to be handled properly with the suggested recommended practices of protection and storage. Any deviations in the suggested practices could have far reaching implications as a single component failure can result in setting aside the entire batch of components leading to cost implications and putting at times the programs in jeopardy.

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