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Effect of Rocket Propulsion Exhaust on Thermophysical Properties of Graphite Nozzle

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Abstract. The present work deals with effect of rocket propulsion system exhaust on thermophysical properties of graphite nozzle. The thermal properties such as thermal diffusivity, specific heat, thermal conductivity, % expansion and coefficient of thermal expansion (CTE) are determined experimentally in the temperature range 100 to 1000 ^{0}C and their analysis is presented along with discussion on measurement methods and effect of rocket propulsion exhaust.

INTRODUCTION

High density graphite are considered to be versatile material, and is used in a wide range of aerospace applications. Graphite with such a range of densities are used in the rocket nozzle having the advantage like very low erosion due to high temperature gas flow, ability to withstand very high temperatures up to 2800^{o}C (inert atmosphere or in vacuum), good thermal shock resistance, self-lubricating, high compressive strength, good chemical resistance, low expansion coefficient at the same time one should be aware of the fact that the thermal expansion of the graphite at high temperature lead to thermal stresses in the material and may be cause for failure of the nozzle as well as complete system**.** Due to their extraordinary properties (e.g. high-temperature stability) graphite materials are often used for high-temperature applications. Many of these applications require the adequate understanding of the thermo physical properties (thermal expansion, specific heat, thermal diffusivity and thermal conductivity). This paper aims to enlighten effect of rocket propulsion system exhaust on the thermo-physical properties of graphite. Thermal diffusivity, specific heat and thermal conductivity of graphite are essential for computational studies related to heat transfer at rocket nozzle as well as erosion studies for high temperature and high velocity gas flowing through nozzle during the operation of rocket propulsion system. To determine thermal properties of graphite, various techniques i.e. guarded heat flow meter, transient plane source and laser flash etc. is used whereas for thermal expansion dilatometer or Thermo-Mechanical Analyser is used. Transient *flash technique* is one of the most popular methods for the determination of thermal properties of materials because it has some advantages like easy to use at high temperatures, non-contact sensing, very fast measurement, absolute for thermal diffusivity measurement etc. Graphite is an anisotropic material; most of the thermo physical properties strongly depend on the measurement direction.

The coefficient of thermal expansion plays the important role in generation of internal stresses in the graphite due to temperature and flux gradients. Yoda and Fujisaki found that there is a direct relationship between the Young's modulus and the coefficient of thermal expansion. To explain this thermal expansion in graphite, Reley derive the theory for quantum thermal expansion of hexagonal lattice using the quantum theory of specific heats which was successfully applied to the graphite single crystal. [1].

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EXPERIMENTAL

Materials and sample preparation

The high density graphite blocks were procured Graphite India, they were machined using lathe to form a disc type sample, having a size of 12.5 mm diameter and 2.2-2.3 mm thick with the accuracy of 10μm. Also the rocket nozzle recovered after the firing of composite propellant for duration~10 seconds was machined to form the same sample as stated above. Figure (a) and (b) shows graphite nozzle before firing (pre-trial) and recovered after firing (post-trial) respectively.

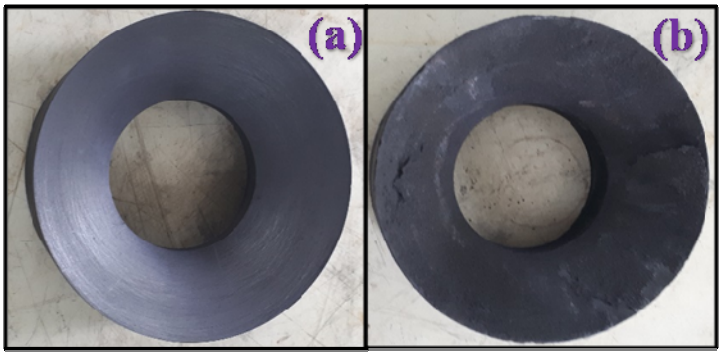

Figure 1. Graphite nozzle (a) before firing (b) recovered after firing

Five samples for each before firing and after firing were prepared. From the same graphite blocks (before firing and recovered after firing) samples for thermal expansion measurement were prepared. Sample for this measurement were cylindrical with dimension of ~50mm length and 6~7mm diameter. Face of the sample were made smooth enough with length measurement accuracy is <30 μm.

Thermal properties measurement

This measurement includes the thermal diffusivity, specific heat, thermal conductivity and thermal expansion measurement with variable temperature. For thermal diffusivity measurement flash method is used whereas thermal expansion measured with dilatometer. In the present study, we have Anter make Flashline 3000 equipment is used to measure thermal conductivity, thermal diffusivity and specific heat simultaneously with varying temperature. The pulsating laser or xenon source energy is incident onto the one side of the sample, which is placed into the sample holder. IR detector (non- contact mode) or fine thermocouple (contact mode) at other end of sample record the thermal perturbation generated in sample with time. This thermal signal received by the detector is further amplified, condition, denoised and passed to data acquisition unit. This data is further processed to obtain the desired results. Flash technique and its theory have been developed by Parker et. al. in 1961[2]. The physical model of the flash technique was developed on the thermal behaviour of an adiabatic (insulated) slab of material, initially at constant temperature, whose one side has been subjected to a short pulse of energy resulting in a temperature rise which forms the base for the measurement. In deriving the mathematical expression from which the thermal diffusivity is obtained, Parker started from the equation of the temperature distribution within a thermally insulated solid of uniform thickness L, as given by Carslaw and Jaeger[3]. Expression for thermal diffusivity achieved is

$$\alpha = \frac{1.38 L^2}{\pi^2 t_{1/2}} \tag{1}$$

Where α is thermal diffusivity, L is thickness of the test sample and $t_{1/2}$ is the time required for the back surface to reach half of the maximum temperature rise. Later on this expression has been corrected for radiation heat losses, finite pulse width etc. by various researchers.

For present measurement, Anter make Unitherm 1000 series dilatometer is used. This experimental set up is in compliance of ASTM E228. It consists of a quartz material push rod which is connected to the digital displacement

transducer. System is calibrated with known reference sapphire for full temperature range and measurement accuracy is found ≤ 5%. Experiments were performed for temperature range RT to 1000 ^{0}C.

RESULTS AND DISCUSSION

The graphite nozzle samples before and after firing were subjected to the flash method and dilatometer methods for determination of its thermal properties. The obtained results are as shown in figure 1.

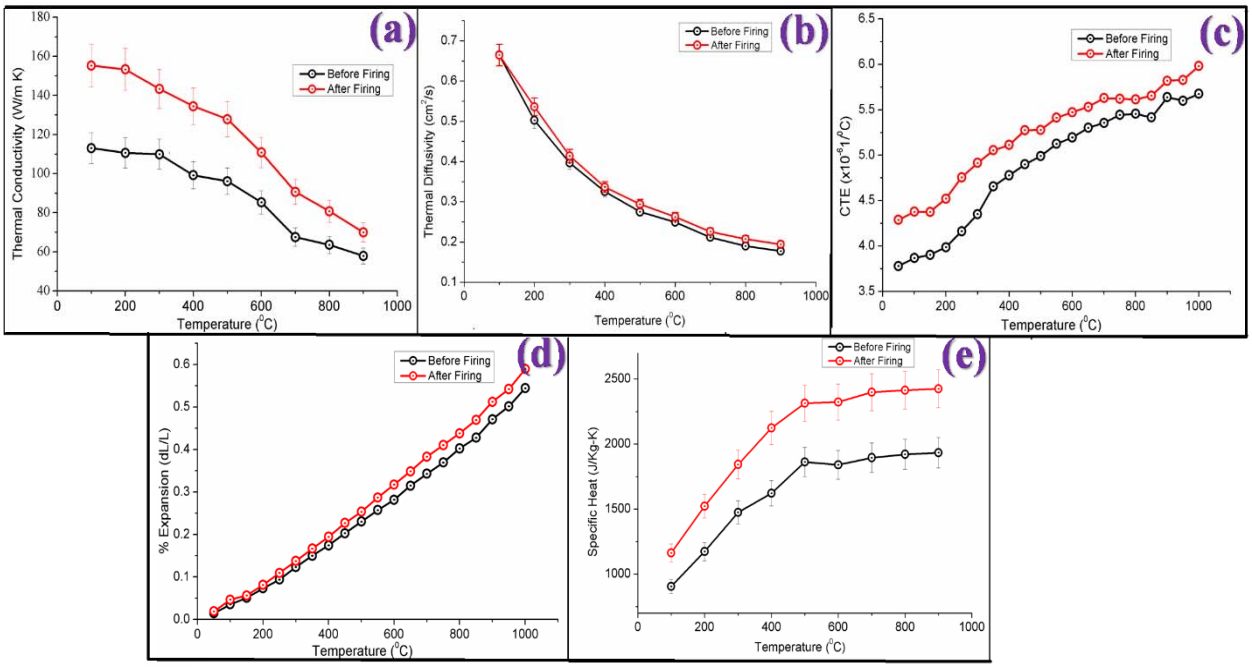

Figure 2. (a) Variation in (a) thermal conductivity (in W/mk) (b)thermal diffusivity (in cm^2 /s) (c) coefficient of thermal Expansion (CTE) , (d) dL/L , (e) specific heat (in J/kg-K) with temperature

Figure 2 (a) shows the variation in thermal conductivity (k) with respect to temperature. It is observed that the thermal conductivity decreases with increase in the temperature for both samples. Average data for both samples shows a deviation < ±7%.These curve shows that post trial graphite sample have higher thermal conductivity than pre-trial. Below 400 ^{0}C temperature, post-trial sample has ~ 40 W/m-K higher thermal conductivity and above the difference between thermal conductivity data decreases and found ~20 W/m-K. Hypothetically it may be due the heat transfer to the graphite material and applied load by the high speed gas which passes through the nozzle during the rocket operation. The thermal conductivity in graphite matrix is majorly dependent on the order in the structure. When we increase the temperature, because of the thermal excitation the stresses are developed in the graphite crystal. These stresses become dominant at higher temperatures. That results into reduction in thermal conductivity. Thermal conductivity is not only dependent on the transport by crystal vibrations but also temperature. Thermal diffusivity results, together with specific heat capacity (Cp) results and density (ρ) values, can be used in many cases to derive the thermal conductivity (λ), according to the relationship: $\lambda=\alpha\ C_p\ \rho$. Fig.2(b) shows that thermal diffusivity of graphite reduces approximately exponentially with increase in the temperature. Also it is evident from the curve that post- trial sample have higher thermal diffusivity than pre-trial sample. The nature of this diffusivity curve is similar to each other with a vertical shift of 0.02 or so. The thermal diffusivity decreases rapidly with increase in the temperature because at higher temperatures higher energy phonons are thermally populated; hence the phonon - phonon scattering becomes stronger the transport of the thermal energy is caused by the phonon or lattice vibrations, the phonon conduction is influenced by the number of phonon active modes and the thermal

resistance by various types of phonon scattering processes such as phonon - phonon scattering, boundary surface scattering and defects scattering.[4].

CTE has to be measured over a particular temperature range. [5] But as the present study is performed to investigate the performance of graphite which is used for rocket population hence the studies were carried out over a wide range (100-1000) ^{0}C as shown in fig. 2(c). It is observed that the CTE increases with increase in the temperature in both the cases and significant difference is observed. CTE of pre-trial sample is relatively lower than that of the post-trial samples. This may be due to the structural modifications which are introduced in the graphite nozzle with flow of the high velocity and high temperature gases. Furthermore the difference between the post-trial and pre-trial CTE curves goes on remains approximately constant with increase in the temperature. Average data for both samples shows a deviation < ±5%. fig. 2(d) shows the % change in length with temperature. At lower temperature both samples have same % expansion but as the temperature increases difference between pre and post-trial samples increases. Post-trial sample have comparatively higher % expansion than pre-trial samples. The specific heat C_v of a material represents the change in energy density U when the temperature changes by 1 K so C_v=dU/dT, where T is the absolute temperature. The specific heat not only determines the thermal energy stored within the body but also it gives the information that how quickly the body cools or heats. At room temperature the specific heat of graphite is C_p=700 Joule kg^{-1} K^{-1} approximately. Interestingly the value of the graphite at the room temperature is ~30% higher than that of diamond because of the higher density of the state at low phonon frequencies given by weak coupling between graphite layers [6].The specific heat of both the samples increases with increase in the temperature as shown in figure 2(e). Average data for both samples shows a deviation < ±6%. Below the 400 ^{0}C temperature, post-trial sample has ~300 J/kg K is higher but above that this difference increases ~500 J/kg K.

CONCLUSION

In the present work, thermos-physical properties of the nozzle graphite materials are studied pre and post trials. A significant variation in these properties is observed for pre and post trials sample. A vertical shift is observed in all measured parameters for the temperature range of 100 to 1000 ^{0}C. Thermal diffusivity decreases exponentially with increase in the temperature for both samples, however maximum vertical shift for post-trial samples in magnitude observed is ~ 0.02 cm^2/s. For specific heat, thermal conductivity, % expansion and CTE difference is variable in different temperature range. These changes can be correlated to structural modifications, by XRD, FESEM and Raman spectrum in further studies, which may occurs due to high pressure, high temperature and high velocity gas flow through the material.

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