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Characterization of an ultra-high temperature ceramic composite (UHTCC) by ultrasonic testing

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Abstract
The ultra high temperature ceramic composites (UHTCC) are of interest for hypersonic vehicle leading edge. The ultrasonic testing is one of the widely used non-destructive testing (NDT) for the material characterization. The aim of investigations described in this article is measurement of the mechanical properties of two ceramic composite by pulse-echo ultrasonic testing. These two samples (ZrB_2-20%ZrC-20%SiC-5%Si_3N_4 and ZrB_2 -20%SiC- 5%Si_3N_4) were fabricated by hot pressing into 25mm diameter and 4mm thickness discs. The measurements were performed using pulse echo ultrasonic technique and ultrasonic transducers with the frequencies 5.0 MHz. It was determined that the velocity of ultrasonic longitudinal and shear waves varies when the frequency is changing. Ultrasound velocity measurements were used to evaluate mechanical properties of UHTCC. These composites were prepared from ZrB_2, SiC, ZrC and Si_3N_4 by ball milling and hot pressing.
Keywords: Aerospace, composite, materials characterization, fracture Mechanics, mechanical properties, wave propagation, ultrasound, velocity.

1. Introduction

Hypersonic flows have been associated with the reentry of orbiting and other high altitude bodies in the atmosphere. Hypersonic flow is the presence of an interaction between the oblique shock wave generated at the leading edge of the body and thermal boundary layer on the surface of the body. Ultra high temperature ceramics composite (UHTCC) are being investiged as a possible approach to overcome all deficiencies.

There are four metallurgical characteristics, which controls all the material properties. These are chemical composition, microstructure, and crystal structure and dislocation density. Material properties can broadly be classified as micro structural properties and mechanical properties. Ultrasonic Testing is the most preferred NDT technique for characterization of material properties. Being volumetric in nature, ultrasonic examination can give an idea about the bulk material properties. Moreover, ultrasonic testing parameters are significantly affected by changes in micro structural or mechanical properties of materials. With the advancement in electronics, these parameters can be measured very accurately to correlate them with various material properties with a reasonable confidence level. Some of the important metallurgical properties that have been correlated with ultrasonic testing parameters are grain size, inclusion content, elastic modulus, hardness, fracture toughness, yield strength, tensile strength, etc. Ultrasonic material characterization has also been used to qualify various processing treatments like precipitation hardening, case hardening, etc. and to assess the damage due to various degradation mechanisms like fatigue, creep, corrosion, hydrogen damage etc.

The scope of material characterization by Ultrasonic is widened by development of Acoustic Microscopes, which produces images similar to the one obtained by optical microscopes. This paper briefly describes the importance of material characterization by non-destructive testing and the affect of material properties on various ultrasonic testing parameters. Ultrasound uses very high frequency sound, higher than 20 kHz (the limit of human hearing), to determine material characteristics of interest such as the presence of cracks, voids, inclusions, porosity, part thickness, weld penetration, and braze and joint integrity. The speeds of wave propagation and energy loss by interactions with material microstructure are key factors in ultrasonic determination of material properties. In the pulse-echo ultrasonic testing technique, an ultrasound transducer generates an ultrasonic pulse and receives its echo. The ultrasonic transducer functions as both transmitter and receiver in one unit. Most ultrasonic transducer units use an electronic pulse to generate a corresponding sound pulse, using the piezoelectric effect. A short, high voltage electric pulse (less than 20 ns in duration, 100-200 V in amplitude) excites a piezoelectric crystal, to generate an ultrasound pulse. The transducer broadcasts the ultrasonic pulse at the surface of the specimen. The ultrasonic pulse travels through the specimen and reflects off the opposite face. The transducer then .listens to the reflected echoes. The ultrasound pulse keeps bouncing off the opposite faces of the specimen, attenuating with time.

2. Experimental procedures

2.1 Materials

The materials investigated are listed in Table 1.They were fabricated by hot pressing. The hot pressed products were discs of 25mm diameter and 4mm thickness.

Table 1. **Materials**

Material	Fabrication	Composition	Designation
ZrB_2-20%ZrC-20%SiC-5%Si_3N_4	Hotpressed, 1850^{0}C, 30 MPa, 30 min graphite die	Reinforcement 20%ZrC-20%SiC-5%Si_3N_4 Matrix- ZrB_2	CMC-4
ZrB_2-20%SiC-5%Si_3N_4	Hotpressed, 1900^{0}C, 30MPa, 30 min graphite die	Reinforcement 20%SiC-5%Si_3N_4 Matrix- ZrB_2	CMC-3

2.2. Procedure

2.2.1. Hardness tests and determination of density

Hardness is done by Vickers indentation method and load is applied 30 kg and 50 kg. Determination of density is based on the Archimedes principle i.e. density is the ratio of weight in air to the weight loss.

2.2.2. Ultrasonic measurements

Elastic modulus is related to the inter-atomic forces and hence indicates maximum attainable strength. There exists a direct mathematical relationship between elastic modulus and ultrasonic longitudinal and shear velocity. These relationships are as follows:

Young's Modulus:

$E = \rho.\ V_T^2.\ (3V_L^2 - 4V_T^2) / (V_L^2 - V_T^2).$

Shear Modulus:

$G = \rho.\ V_T^2.$

Bulk Modulus:

$B = \rho.\ (V_L^2 - 4.\ (V_T^2) / 3).$

Poisson's ratio:

$\upsilon = (V_L^2 - 2.\ V_T^2) / (2.V_L^2 - 2.\ V_T^2).$

Fracture toughness K_C is given by:

$K_C = \sqrt{E'}.\ G_c,$

E' is the Young's modulus G_c is the strain energy release factor.

2.2.3. Metallographic investigation

After surface preparation, the specimens were examined under the optical microscope with the magnifications of x100 and x200, etching in 10ml glycerin+ 10 ml HNO_3 + 10 ml HCl + 0.1 ml HF.

3. Results and discussion

The accuracy of any ultrasonic measurement is only as good as the accuracy and care with which the gage has been calibrated. All quality ultrasonic gages provide a method for calibrating for the sound velocity and zero offset appropriate for the application at hand. It is essential that this calibration be performed and periodically checked in accordance with the manufacturer's instructions. Sound velocity must always be set with respect to the material being measured .Zero offset is usually related to the type of transducer, transducer cable length and mode of measurement being used.

Result of ultrasonic testing is given in Table 2

Table 2. **Ultrasonic testing**

Material	V_L(m/s)	V_T(m/s)	E(GPa)	Poisson's ratio
CMC-3	9553	6156	452	0.15
CMC-4	9009	5682	387	0.17

Result of Hardness and fracture toughness is given in Table 3 (Taking E=452GPa & 387GPa).

Table 3. **Hardness & Fracture toughness (Charles, Nlihira, Anstis and Laugier)**

Material	H (MPa)	C µm	K_{IC} MPa - m$^{1/2}$	K_{IC} MPa - m$^{1/2}$	K_{IC} MPa - m$^{1/2}$	K_{IC} MPa - m$^{1/2}$	Load
CMC-3	25.22	307	4	5	4	3	30kg
CMC-3	21.51	445	3	5	4	3	50kg
CMC-4	32.25	256	5	5	4	3	30kg
CMC-4	30.18	416	4	5	5	4	50kg

Density is calculated based on Archimedes's principle is given in Table 4

Table 4. **Density measurement**

Material	Hot pressing condition	Dimension (mm × mm)	Density (gm/cc)
CMC-3	Hot pressed, 1900^{0}C, 30MPa, 30 min graphite die	24.32 × 4.92	5.21
CMC-4	Hotpressed, 1850^{0}C, 30 MPa, 30min graphite die	24.52 × 5	5.13

Ultrasonic testing for material characterization can not play a vital role in quality assurance during in-manufacture inspection but can serve as a powerful tool for life prediction technology during in-service inspection, residual life assessment and plant life extension. There are however few difficulties which are encountered during ultrasonic testing for material characterization. There is no one to one mapping between ultrasonic parameters and microstructural / mechanical properties. Microstructural properties, which control the mechanical properties, affect the ultrasonic propagation factors differently. In order to establish any correlation between mechanical properties and ultrasonic parameters the effects of various microstructural properties on ultrasonic testing parameters must be separated.

4. Conclusion

Ultrasonic testing is traditionally used for flaw detection and characterization. The spectrum of ultrasonic testing applications is widened by its use for material characterization. With the advancement in electronics and digital technology, ultrasonic testing parameters, which are affected by changes in material properties, can be measured with high accuracy to provide a reasonable confidence level.

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Superaukštųjų temperatūrų keraminių kompozitų (UHTCC) apibūdinimas remiantis ultragarsiniais bandymais

Reziumė

Superaukštųjų temperatūrų keraminiai kompozitai naudojami viršgarsinių lėktuvų priekinėse briaunose. Ultragarsiniai bandymai labai tinkami medžiagoms apibūdinti neardomuoju būdu. Straipsnyje aprašytų tyrimų tikslas buvo dviejų keraminių kompozitų mechaninių savybių matavimas impulsiniu ultragarsiniu aido metodu. Tam karšto presavimo būdu buvo pagaminti du 25 mm skersmens ir 4 mm storio bandiniai (ZrB_2-20%ZrC-20%SiC-5%Si_3N_4 ir ZrB_2 -20%SiC- 5%Si_3N_4). Matavimai buvo atlikti impulsiniu aido metodu, naudojant 5 MHz dažnio ultragarsinius keitiklius. Nustatyta, kad išilginių ir skersinių ultragarso bangų greitis priklauso nuo dažnio.

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