

REVIEW OF AVAILABLE DATA FOR ASSESSMENT OF NON-INTRUSIVE DIAGNOSTIC METHODS FOR INTERNAL MATERIAL PROCESS DIAGNOSTICS -EXECUTIVE SUMMARY

NIDIAB – ESA Contract 4000112225/14/NL/MV

Philippe Reynier

ISA TN-05-2015

NOVEMBER 2015

Ingénierie et Systèmes Avancés Bât. O – Parc Cadéra	Visa	Ph Reynier
12, rue Ariane F-33700 Mérignac	Version n°	1
Tel: +33 (0) 556479274 Fax: +33 (0) 556473148	Date	4 th November 2015

CONTENTS

PAGE N°

1	NOMENCLATURE	5
2	SUMMARY	6
3	INTRODUCTION	7
4	ACTIVITY OVERVIEW	8
5	ACTIVITY ACHIEVEMENTS	9
5.3 5.4	Physical processes Survey of non intrusive techniques Technology assessment 1 Terahertz microwave imaging 2 X-ray, γ-ray, and neutron absorption Ultrasound – Identified issues	10 10 10 11 11
6	ROADMAP FOR TECHNOLOGY DEVELOPMENT	13
6.1 6.2 7	Selected methods Roadmap	13
8	REFERENCES	15

LIST OF FIGURES

Figure 1: Work breakdown structure	8
Figure 2: Phenomena occurring during ablation of porous phenolic ablative materials [3]	
Figure 4: Set up for ultrasound mounted on an ICP torch []	11

1 NOMENCLATURE

Acronyms

PWT Plasma Wind-Tunnel

2 SUMMARY

This is the executive summary for the GSP activity "Review of available data for assessment of nonintrusive diagnostic methods for internal material process diagnostics" led by ISA under ESA contract 4000112225.

3 INTRODUCTION

The purpose of this GSP activity was to assess the capabilities of non-intrusive diagnostic techniques for internal measurements in ablative materials during PWT tests. This initial steps were to identify the measurements to be performed in relation with high-speed Earth re-entry, and to identify the accessible parameters as well as related diagnostics techniques. For this objective an extensive review of the possible measurement methods, with a focus on non-intrusive diagnostics, has been undertaken. The review has indicated that the most promising methods were:

- Ultrasound;
- Terahertz microwave;
- Neutron absorption;
- X-ray and γ-ray scanning.

These techniques were selected for further investigation and finally the most promising (ultrasound and X-ray scanning) retained for the development of an experimental set up accounting for hardware availability for future demonstration in PWT.

It has to be noted that in the frame of the activity measurements of the transmitivity of ablative material samples in the terahertz microwave range has been achieved.

4 ACTIVITY OVERVIEW



Figure 1: Work breakdown structure

5 ACTIVITY ACHIEVEMENTS

5.1 Physical processes

Ablative heat shield materials are currently the only solution for a safe Earth re-entry when high-speed entry, e.g. hyperbolic sample return, is considered [1]. The different processes related to ablation are summarized in Figure 2. Many different numerical tools are used to investigate the highly coupled phenomena occurring in an ablation environment [2]. Current research focuses on the detailed understanding of the in-depth material processes with coupling to the flow-field and radiation including non-equilibrium chemistry [3-4-5].





Most of the experimental work focuses on the performance analysis of ablative materials using indepth thermocouples [6]. As a consequence, sophisticated analysis tools have been developed in order to cover pyrolysis, nitridation, oxidation, and surface recession or spallation [4]. On the other hand, for the analysis of ablative materials in ground testing facilities, sophisticated non-intrusive diagnostics are available to analyse the boundary layer flow [7,8]. The next step for the better understanding of the processes is be the analysis of in-depth the ablation phenomena, i.e. surface recession, spallation, change of morphology, change of composition, and optical properties. For this objective, the development and application of non-intrusive diagnostic measurement techniques is needed. The measurements required for improving the state-of-the-are when testing ablative samples are:

- 1. Temperature profiles;
- 2. Internal pressure;
- 3. Heat flux;
- 4. Recession rate;
- 5. Pyrolysis gas composition (internal);
- 6. Pyrolysis gas mass flow;
- 7. Char depth;
- 8. Surface catalycity;
- 9. Radiation (internal, external);
- 10. Spallation;
- 11. Density (solid, gas, total);
- 12. Sublimation (if occurring);
- 13. Volumetric oxidation/nitridation;
- 14. Microstructure, real time imaging;

5.2 Survey of non intrusive techniques

An extensive survey of non-intrusive experimental techniques has been undertaken in relation with plasma wind-tunnel tests (PWT) of ablative materials accounting for the efforts performed in the previous work package [9]. The techniques in use for the development of non-space applications such as medicine, energy, chemical engineering and electronics have been considered. This survey takes advantages of a previous effort [10] done for the experimental investigation of tank sloshing. Here, a particular effort has been dedicated to electric and ultrasound techniques since these techniques are cheap and being developed for TPS investigation. Other methods based on radiation, microwave, magnetic, and holographic approaches have been also considered in the current effort.

The different techniques have been assessed and some potential issues identified. When possible, the potentialities and drawbacks for the different methods have been summarized with for objective their application during PWT tests. The existing background on PWT testing or ablator instrumentation has also been considered. This has allowed the evaluation of the different methods and a preliminary trade-off was proposed to the Agency: Terahertz microwave imaging, ultrasound tomography, radiation (X-ray, γ -ray, and neutron absorption) scanning have been retained for further investigation.

5.3 Technology assessment

5.3.1 Terahertz microwave imaging

To investigate terahertz imaging capabilities, transmitivity of ablative material samples has to be studied. Series of tests have been carried out on charred and virgin samples for small material thickness (1 and 3 mm) in different research laboratories for a wide range of frequencies. More would be required to investigate the terahertz range, since there is a gap from 0.08 up to 0.1 THz, but too much efforts would be necessary. From the current state-of-the art Terahertz/microwave imaging is

found unsuitable for the targeted objectives due to the low maturity level of this technique and to the extensive efforts that would be necessary to improve the current state of the art.

5.3.2 X-ray, γ-ray, and neutron absorption

Further investigation has shown that a large neutron source would be necessary for neutron imaging. A dedicated facility would be needed, with a large size, as a consequence this could not be used in a PWT.

For the current application, the issue with γ -ray, beside with the safety aspects, is the high level of energy requires by the technique. The available hardware require an energy higher than 60 keV, such level is too high for measuring density gradients, since material details can be observed only with energy levels below 20 keV.

X-ray scanning is currently used to characterize the internal structure of ceramics and composites. So far, no results on its application during PWT tests of ablative materials has been found. However, there is no identified drawback for using this method during PWT, and its application would be attractive. As a consequence X-ray sources and detectors have been studied and potential suppliers sourced by IRS.



Figure 3: Set up for ultrasound mounted on an ICP torch [11]

5.4 Ultrasound – Identified issues

Experimental sets up have been already designed (see Figure 4) and test campaigns conducted using ultrasound sensors. To determine the surface recession the time of flight for a sensor located on the rear surface of a sample has to be estimated. For this objective, the sound velocity in the material has to be known as well as the influence of temperature on it. If the material, temperature can be determined, the sound velocity also, then the time of flight can be determined as done by Llyod [12]. This is an important issue and before testing samples a good knowledge of their properties (elasticity, Young modulus) will have to be determined to asses the variation of sound velocity within the material as function of temperature.

Capabilities for in-depth measurements is well known, as a consequence you can have an in-depth characterization of the material. This is extensively done in echography. Echography is used in medicine (on an image you can see bones, nerves, ...), but also to check for internal failure in pipes or tanks (for example). The reflection depends on the inner properties of the material (ultrasound impedance).

In Europe research activities on ultrasound testing are conducted in numerous universities. For PWT testing, the best option for the next step is to use commercially available hardware and software (for image processing). Companies producing hardware have been also identified, but there is apparently nothing on shelf ready to be used for material testing in PWT. A price breakdown for an ultrasound kit for material characterization has been provided by a potential supplier. However, further knowledge on the material itself would be needed and a sample provided to have the best possible adaptation of the kit, as well as a sample holder design for developing a dedicated kit for PWT tests. This would, of course, increase the cost.

6 ROADMAP FOR TECHNOLOGY DEVELOPMENT

6.1 Selected methods

According to the study findings, the most promising methods are;

- Ultrasound tomography
- X-ray scanning.

Their application would allow to determine surface recession and internal phenomena (cracking, potential fractures in the microstructure of the material, evolution of pyrolysis).

6.2 Roadmap

The following steps have been identified to ensure the development of the selected methods up to an use in high enthalpy facilities:

- Design of a set-up that includes measurements with the selected techniques;
- Identification of necessary hardware and software;
- Identification and selection of suppliers Procurements;
- Test campaign on virgin and char samples;
- Test in PWT.

7 RECOMMENDATIONS FOR FUTURE ACTIVITIES

The following recommendations can be given for future studies dedicated to the application of non intrusive diagnostic techniques for testing ablative material samples in high enthalpy facilities:

- Among the reviewed techniques X-ray scanning and ultrasound tomography are the most promising for the targeted application.
- Recommendation is to go further in this direction with the applications of the techniques to PWT.
- Due to the issue related to sound velocity for ultrasound, material properties such as elasticity (Young modulus) have to be determined.
- Due to the fact that the application of such techniques on PWT is a pioneering work; it is strongly recommended to use them conjunctly for having cross-check between the measurements.

8 **REFERENCES**

- [1] Johnston, C. O., Gnoffo, P. A., and Sutton, K., Influence of ablation on radiative heating for Earth entry, Journal of Spacecraft and Rockets, Vol. 46(3), pp. 481-491, May-June 2009.
- [2] Lachaud, J., Magin, T. E., Cozmuta, I., and Mansour, N. N., A short review of ablative-material response models and simulation tools, Proceedings, Brugge, Belgium, 9-12 May 2011.
- [3] Lachaud, J., and Mansour, N. N., Porous-material analysis toolbox based on OpenFoam and applications, Journal of Thermophysics and Heat Transfer, Vol. 28(2), pp. 191-202, April-June 2014.
- [4] Milos, F. S., and Chen, Y.-K., Ablation and thermal response property model validation for phenolic impregnated carbon ablator, Journal of Spacecraft and Rockets, Vol. 47(5), pp. 786-805, Sept.-Oct. 2010.
- [5] Stern, E. C., Nompelis, I., Schwartzentruber, T. E., and Candler, G. V., Microscale simulations of porous materials: Application to permeability, AIAA Paper 2014-2247.
- [6] White, T., Cozmuta, I., Santos, J. A., Laub, B., and Mahzari, M., Proposed analysis for Mars Science Laboratory heat shield sensor plug flight data, AIAA Paper 2011-3957.
- [7] Loehle, S., Staebler, T., Reimer, T., and Cefalu, A., Photogrammetric surface analysis of ablation processes in high enthalpy air plasma flow, AIAA Paper 2014-2248, 2014.
- [8] Hermann, T., Zander, F., Fulge, H., Löhle, S., and Fasoulas, S., Experimental setup for vacuum ultraviolet spectroscopy for Earth re-entry testing, AIAA Paper 2014-2536, 2014.
- [9] Eberhart, M., Zander, F., and Löhle, S., Technical Note 1 : Non-intrusive diagnostics for ablation, IRS-15-P04, IRS, Stuttgart, 24 April 2015.
- [10] Poette, C., & Reynier, Ph., Evaluation of 3D mapping experimental non-intrusive methods for multiphase flows, International Journal of Multiphysics, Vol. 8(1), pp. 63-84, 2014.
- [11] Hurley, D. A., Huston, D. R., Fletcher, D. G., and Owens, W. P., Thermal protection system monitoring using acoustic emission, Nondestructive Characterization for Composite Materials, Wu, H. F. Ed., Proc. of SPIE Vol. 7983, 2011.
- [12] Lloyd, J. A., Stackpole, M., Venkatapathy, E., and Yuhas, D. E., A new non-intrusive, ultrasonic TPS recession measurement needed to determine the thermal structure of the upper atmosphere of Venus, Saturn, Uranus, or Neptune, Paper 1111, International Workshop on Instrumentation for Planetary Missions, (IPM-2012), Greebelt, Maryland, 10-12 Oct. 2012.