	Name of the PhD candidate: **Lucile BARKA** Title of the PhD thesis: **Study of the survivability of space debris during their atmospheric re-entry phase (oxidation, ablation, emissivity)** Dates (start/end): 01/10/15 – 30/09/18
Supervisors	Marianne Balat-Pichelin, PROMES-CNRS
Other contributors	Co-funding CNES Toulouse & SOLSTICE

Context and objectives

In order to mitigate debris in orbit and to avoid dramatic collisions on Earth after atmospheric re-entry at its end-of-life, the spacecraft missions have to take into account the influence of atmospheric re-entry conditions (high temperature, air plasma and low pressure) on the spacecraft survivability. CNES has developed the DEBRISK tool to study the survivability of space debris but databases currently available in this tool are limited for oxidation kinetics laws and for thermal radiative properties (total hemispherical emissivity) of materials at high temperature. To increase the calculation accuracy of the DEBRISK tool, CNES collaborates with PROMES-CNRS laboratory to perform oxidation in atmospheric re-entry conditions and to measure the emissivity of materials at high temperature.

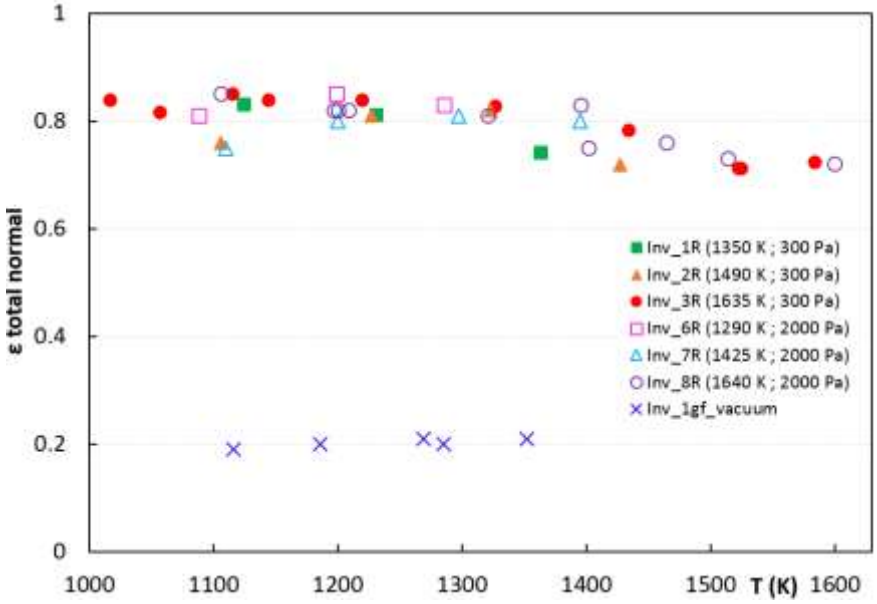
Approach

In this way, oxidation in air plasma conditions was studied on metallic alloys such as TA6V, SS 316L & 304L and Invar 36 in order to obtain degradation kinetic laws at high temperature (up to the melting point of the alloys) and for short time duration (500 s max), using the MESOX (Moyen d'Essai Solaire d'OXydation) device implemented at the focus of the 6 kW solar furnace. Moreover, measurements of the total (0.6-40 µm) directional emissivity – the hemispherical value being obtained by integration of the directional data – at high temperature were carried out on the same virgin alloys – in high vacuum to avoid oxidation – and under controlled pressure on samples pre-oxidized in air plasma conditions, and in situ in standard air for comparison. Emissivity measurements were performed using the unique MEDIASE (Moyen d'Essai et de Diagnostic en Ambiance Spatiale Extrême) device. The important impact of oxidation in air plasma on emissivity was observed by different characterization techniques using Scanning Electron Microscopy (SEM), 3D profilometry, X-Ray Diffraction (XRD) and X-ray Photoelectron Spectroscopy (XPS).

Main results

Kinetic laws for the oxidation in air plasma conditions and emissivity data at very high temperature were obtained for all the materials studied. For example, SS 316L and Invar 36 were oxidized in air plasma conditions (80% of dissociated oxygen) at high temperature under 300 and 2000 Pa for duration up to 500 s. With the increasing temperature, the mass gain is clearly huge for Invar 36 compared to SS 316L. Laws with their respective activation energies were determined for the evolution of the mass gain versus temperature (in the range 1000-1670 K) and versus time according to the total air pressure. Oxides formed on the surface of these two materials indicate the presence of hematite Fe_2O_3 and magnetite Fe_3O_4 according to the temperature level. It was shown that the composition changed for Invar 36 with the increasing temperature, Fe_3O_4 being predominant at higher temperatures. Emissivity measurements were performed on virgin surfaces in high vacuum and also on pre-oxidized samples in air plasma conditions and oxidized in situ in standard air under 300 and 2000 Pa at high temperature. For example, a significant increase of the total normal emissivity by a factor 3 was observed for the SS 316L pre-oxidized samples (0.85-0.95) compared to virgin ones (0.30-0.25) and by a factor 4 for Invar 36. Moreover, in the case of Invar 36, a different trend is observed according to the temperature, with an emissivity stable around 0.80 up to 1400 K, followed by a decrease around 1450 K (0.75) and again a stable value (0.70) from 1500 K up to the melting point (1700 K). This decreasing was correlated mainly to the smoothing of the surface roughness of the sample and in a lower proportion to the chemical composition of the oxide layers. Experiments carried out in standard air have given different data that are lower than the ones measured on pre-oxidized samples in air plasma conditions.

So, the emissivity data obtained on pre-oxidized samples are mandatory for their implementation in the DEBRISK code to better model the mass reaching the ground taking into account the increasing thermal losses by radiation due to oxidation.



Evolution of the total normal emissivity versus temperature for the non-oxidized Inv_1gf sample measured in high vacuum and for several pre-oxidized Invar samples (Inv_xR) at 300 and 2000 Pa in air plasma conditions

Publications

- BARKA L., BALAT-PICHELIN M., SANS J.L., BECHE E., Oxidation and emissivity of Invar 36 alloy in air plasma at high temperature, submitted to *Journal of Alloys and Compounds,* March 2018

International conferences

- BALAT-PICHELIN M., BARKA L., ANNALORO J., Oxidation and emissivity of space debris materials (ceramic and metallic alloys) during their atmospheric entry, *High Temperature Materials Chemistry HTMC 15,* Orléans (France), 29 mar-1 april 2016

- BALAT-PICHELIN M., BARKA L., ANNALORO J., OMALY P., Atmospheric entry of space debris: oxidation and emissivity data for model implementation, *8th International Association for the Advancement of Space Safety IAASS conference*, Melbourne (Fl, USA), 18-20 May 2016, "Safety first, safety for all" proceed. 526-534.

- BALAT-PICHELIN M., BARKA L., SANS J.L., ANNALORO J., Atmospheric entry of space debris: influence of oxidation and emissivity in the calculation of the spacecraft survivability, *14th European Conference on Spacecraft Structures, Materials and Environmental Testing ECSSMET 2016*, Toulouse (France), 27-30 Sept 2016

- BARKA L., BALAT-PICHELIN M., SANS J.L., ANNALORO J., OMALY P., Influence of oxidation and emissivity for metallic alloys space debris during their atmospheric entry, *7th European Conference on Space debris*, ESA/ESOC, Darmstadt (Germany), 18-21 April 2017

- BARKA L., BALAT-PICHELIN M., BECHE E., SANS J.L., ANNALORO J., OMALY P., Oxidation laws and emissivity data at high temperature for implementation in DEBRISK code, *9th International Association for the Advancement of Space Safety IAASS conference*, Toulouse (France), 18-20 Oct 2017, Proc. ISBN 978-90-828378-0-3, 31-40.

- BALAT-PICHELIN M., BARKA L., BULTEL A., ANNALORO J., Characterization of space debris materials during their atmospheric entry, *14th International Symposium on Materials in the Space Environment ISMSE 14*, Biarritz (France), 1-5 Oct. 2018.