	Experiment title: New soldering alloy without lead for Microelectronics applications: in situ X-ray microtomography at high temperature for reliability testing	Experiment number: MA-5289
Beamline: ID19	**Date of experiment**: from: 01/04/2022 to: 04/04/2022	**Date of report**: 27/10/2022
Shifts: 9	**Local contact(s)**: Marta Majkut	

Names and affiliations of applicants (* indicates experimentalists):

Raphaël Pesci, Marc Wary: ENSAM-Arts et Métiers Sciences and Technologies, laboratory LEM3 UMR CNRS 7539, 4 Rue Augustin Fresnel, 57078 Metz, France

Mathilde Buret, Mélanie Mathon: Inventec Performance Chemicals, 26 Rue des Coulons, 94360 Bry-sur-Marne, France

Aim of the experiment and scientific background

Two lead-free substitution alloys have been developed. We aimed to determine how they perform under real brazing: in particular the integrity of the interface with the substrate (presence of cracks or not) and when and how voids occur in the joint, because these are key points for industrial applications. The number and the localization of voids are important since a certain level of voids can help for residual stress relief in the joint, but too many of them can lead to dramatic failure; the thermal expansion coefficient of the solder alloy has been chosen to be very close to that of the most used substrates, but the evolution of the microstructure at high temperature and the formation of intermetallics can be prohibitive for service life. It was thus crucial to carry out in situ X-ray microtomography during heating and cooling/solidification to detect the presence of voids and cracks in the solder joints.

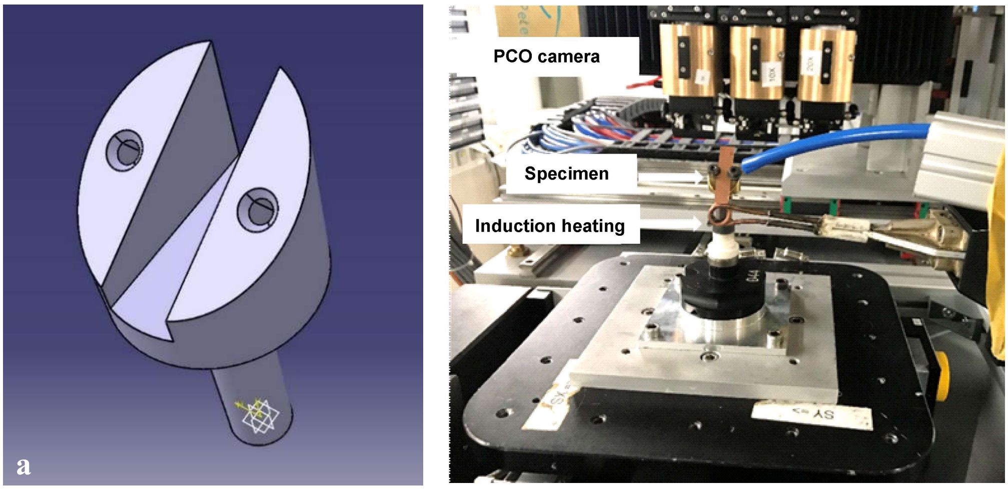

Figure 1- a: holder for continuous radiography - b: experimental device at ESRF (ID19 beamline)

Experimental method

A specific holder has been designed to be able to observe the evolution of each solder alloy during heating and cooling. With an inclination of 45° (figure 1a), it allows the X-ray beam to pass through and to collect the absorption images continuously with high acquisition frequency (continuous radiography). The 1mm thick substrate is placed on it after depositing a pad of solder paste (powder+flux) by screen printing through a stainless steel stencil, then the holder is induction-heated until melting and cooled (figure 1b).
Once the soldering has been made, the substrate is machined in order to release a cylinder 1mm in diameter with the solder alloy at the top (figure 2). It is thus possible to use standard microtomography (80keV, 3000 images with a step of 0.12°) to get a 3D reconstructed volume with a resolution lower than 1µm.

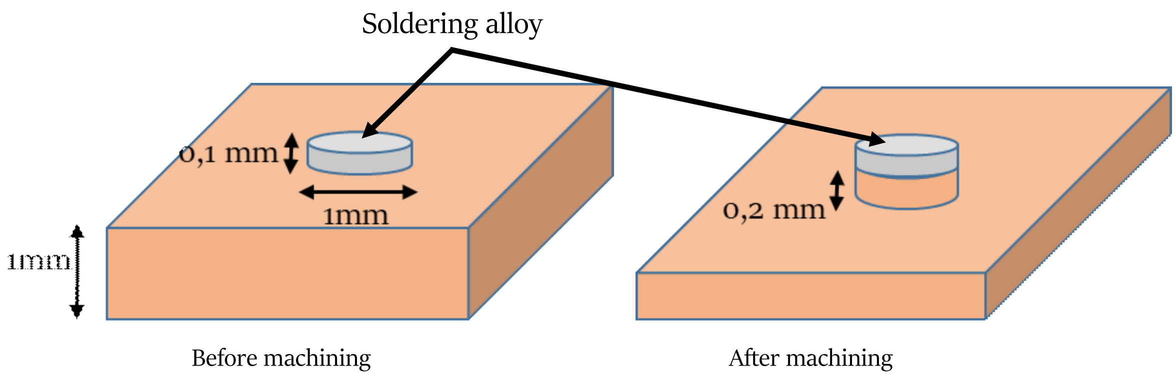

Figure 2- Specimen used for standard microtomography (3D reconstruction)

Results

Several alloy/substrate/flux combinations were tested with a total of 67 continuous radiographs (videos) and 81 tomographic 3D reconstructions; protective gas was also considered for some specimens. Continuous radiography made it possible to observe the evolution of the phenomena that occur during soldering. On the images of figure 3, one can observe in particular the formation of gas bubbles/voids linked to the evaporation of the flow and their evolution during the heating and cooling cycle (growth, coalescence, dissipation at the surface).

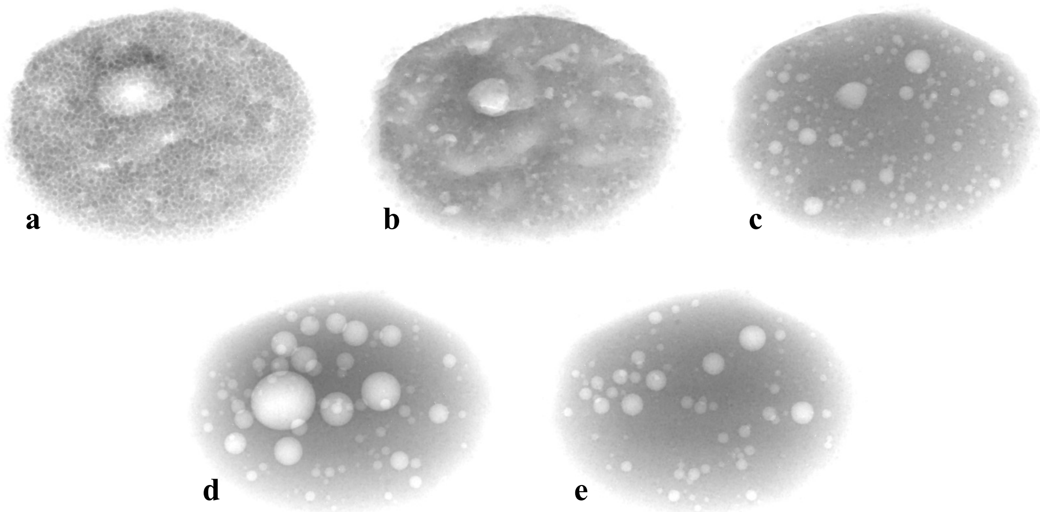

Figure 3- Evolution of the SnSbCu soldering alloy during the heating + cooling cycle (copper substrate)
a: initial solder paste (powder+flux) - b: melting - c and d: evolution of gas bubbles/voids
- e: final state after cooling

The interaction between the soldering alloy and the substrate can also lead to the formation of intermetallic compounds or defects such as cracks at the interface, as observed using a scanning electron microscope (figure 4).

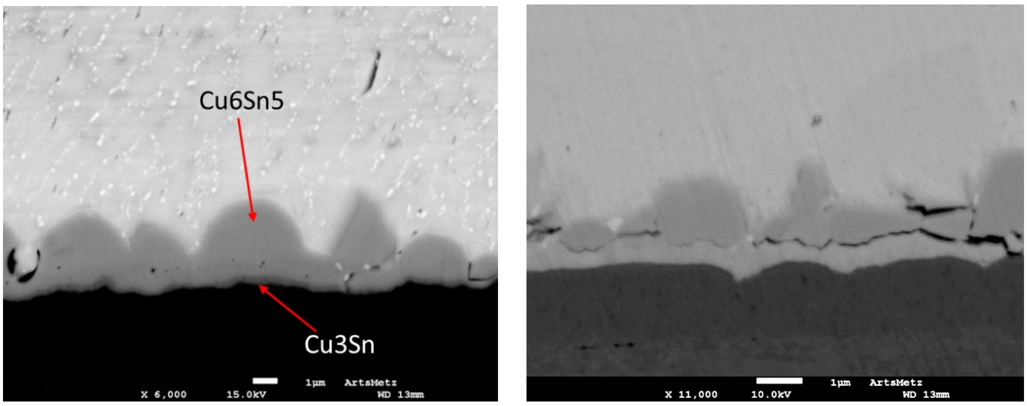

Figure 4- Intermetallic compounds (left) and cracking (right) at the interface between the soldering alloy and the substrate

The 3D imaging resulting from the tomographic analyzes was carried out using the Tomwer software on the ESRF cluster (figure 5). The aim is to get the shape of the solder to see if the wettability is good, to detect the intermetallic compounds when possible and to determine the quantity of voids, their position and size since all have an influence on the quality of the solder and the final properties during service life.

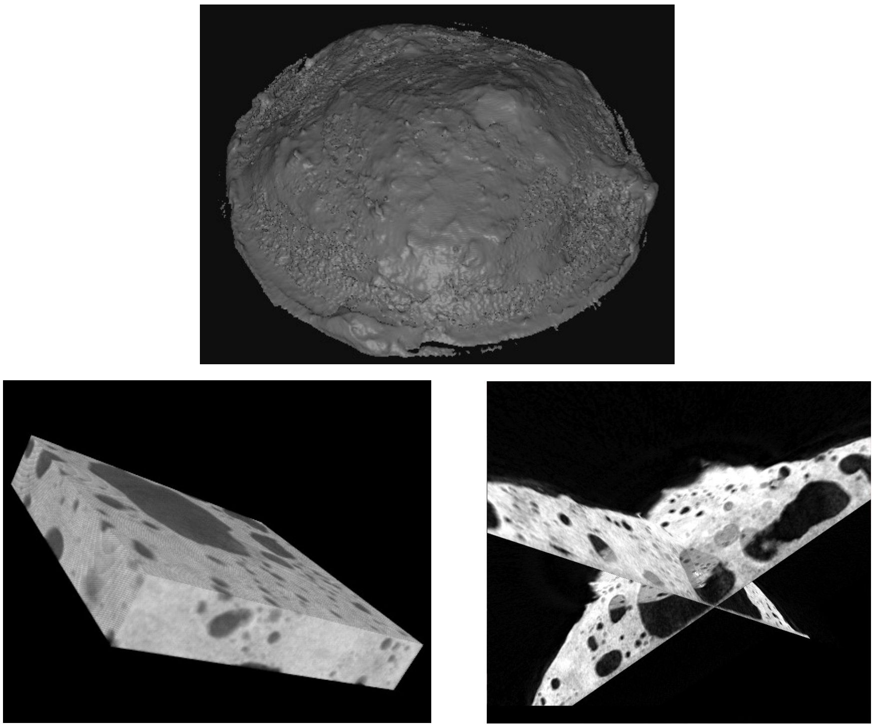

Figure 5- 3D reconstructions and cross-sections after processing by Tomwer software

Conclusions

These experiments coupling X-ray radiography and microtomography at ESRF made it possible to collect a substantial volume of data and to better understand some phenomena never before observed in situ. All these data must now be processed and analyzed for each experimental condition tested (soldering alloy, substrate, protective gas…) in order to validate the properties of the 2 lead-free substitution alloys tested and publish the results in international scientific journals.