Physico-chemistry of zirconia to titanium brazing using pure gold: wetting and interfacial reactivity

Marie FISCHER

Supervisors: Fiqiri Hodaj and Valérie Chaumat

Friday, March 25th 2022 – 9:00 a.m.

Amphithéâtre Ampère (GreEn-E)

Jury:
Marie-Laurence GIORGI, PR. DES UNIVERSITES CentraleSupélec, Rapporteur
Luis Filippe MALHEIROS, PR. Université de Porto, Rapporteur
Christophe GIRARDEAUX, PR.DES UNIVERSITES Université d'Aix-Marseille, Examinateur
Jean-Michel MISSIAEN, PR. DES UNIVERSITES Grenoble INP, Examinateur

Abstract: This work deals with physico-chemical aspects of zirconia-to-titanium joining by brazing for a biomedical application (cochlear implant), mainly supported by microscopy analyses (SEM, FEG-SEM, TEM) of interfacial reaction products.

In a first section, we performed an experimental study of wetting and interfacial reactivity of zirconia and titanium substrates, using the «sessile drop» and «dispensed drop» methods. First, wetting of zirconia by the biocompatible active brazing alloys (Ag-A, Au-Sn-A and Au-A, A being Ti or Zr active element) was studied, in the temperature range 1066°C - 1270°C. Zirconia is well wetted by all these selected alloys. A comprehensive study was conducted with the Au-xTi ($0.6 < x < 4$ wt%) alloys, as they are the best candidates for zirconia-to-titanium brazing in terms of interfacial reactivity. Indeed, the wettable TixOy reaction product layer formed at the ZrO2 / alloy interface is homogeneous, continuous and thin (0.3 – 4 µm). Then, wetting study of titanium substrate with pure gold was performed in the temperature range 1077 – 1125°C. The non-reactive wetting of Au on Ti, observed during the first ten milliseconds, is followed by a much longer reactive spreading stage occurring by dissolution and formation of a reaction product layer at the interface. Good wetting of Ti is obtained, whatever the surface state of the substrate (with or without native oxide).

In a second section, ZrO2 / Ti brazing with pure gold was investigated. The joint is created thanks to the formation of reaction products at both interfaces. At Ti / Au interface, four Ti-Au intermetallic compounds (Ti3Au, TiAu, TiAu2 and TiAu4) and the δ-Au solid solution are observed. Ti dissolution and diffusion into liquid Au lead to the formation of a wettable TixOy layer at the ZrO2 / Au interface. Oxygen is provided by zirconia, becoming sub-stoichiometric near the joint. The impact of several factors such as filler metal thickness, brazing temperature, holding time and oxygen partial pressure was investigated in order to propose optimum brazing parameters. This brazing study was supported by tensile tests, leading to an average rupture stress of about 56 MPa. For the first time, a relation between the oxygen partial pressure in the brazing furnace and the mechanical properties of the joint was experimentally established.