




	Experiment title: RESIDUAL STRESS DETERMINATION IN HYDROXYAPATITE PLASMA-SPRAY COATINGS	Experiment number: HS-988
Beamline: ID15A	Date of experiment: from: 11 th November to: 18 th November 1999	Date of report: 29/2/2000
Shifts:	Local contact(s): Klaus-Dieter Liss, Thierry d'Almeida	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Jean-Michel Sprauel*, Adele Carrad  *, Laurent Barrallier*, Agnès Fabre* * Ecole Nationale Supérieure d'Arts et Métiers (E.N.S.A.M.) Laboratoire MECASURF 2, Cours des Arts et Métiers - F-13617 AIX EN PROVENCE Cedex 01		

Report:

High-energy synchrotron measurements are a promising tool for the investigation of residual stresses in plasma spray coatings on metal substrate.

Hydroxyapatite (HA) coatings are used on metallic prothesis to increase the bone - metal interface strength and to improve the device biocompatibility.

The experiments have been performed at Beamline ID15A using the white beam (energy range from 50 to 150 keV). The gauge volume was limited to $60 \times 700 \times 100 \mu\text{m}^3$ by slits in the primary as well as in the diffracted beam. The diffracted intensities were recorded by means of an energy dispersive Germanium detector.

The specimens were two palladium alloy substrates with and without Hydroxyapatite coating ($\text{Ca}_5(\text{PO}_4)_3\text{OH}$). The dimensions of the uncoated sample was $60 \times 20 \times 2 \text{ mm}^3$ and for HA coated $60 \times 20 \times 3.35 \text{ mm}^3$.

Because of the great difference existing between lattice parameter of Hydroxyapatite and the Palladium, the measurements have been focused on the evaluation of the stresses in the first layers of the base metal. The {220}, {311}, {222}, {331}, {420}, {422}, {333}, {440}, {620}, {642} and {731} reflections of the FCC phase of Palladium have been analysed for that purpose by using diffraction angle $2\theta = 5^\circ$. (*Fig. 1*)

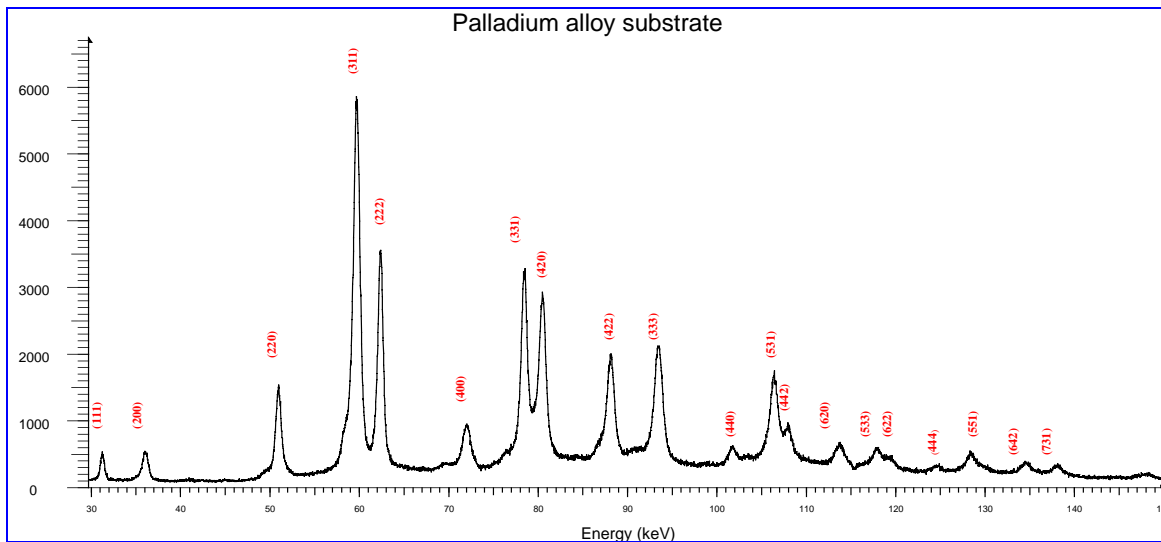


Figure 1

The $\sin^2\psi$ method has been used to evaluate the stresses in the longitudinal directions of the sample. These measurements were carried out in χ mode with four incidences (0° , -30° , -45° and 37°).

The depth affected by the stresses is of the same order of magnitude than the size of the X-rays beam. It is therefore necessary to localise very precisely the true position of the diffracting volume inside the sample. This is obtained through a strain scanning across the studied interface. The results show very strong absorption effects. For this reason the experiment was modelled through a Monte Carlo simulation method which allows to define the evolution of diffracted intensity versus depth and also to evaluate the true mean position of the diffracting volume. (Figure 2) The true position of analysed volume is then derived from these curves. The reliability of this method is better than $2\ \mu\text{m}$.

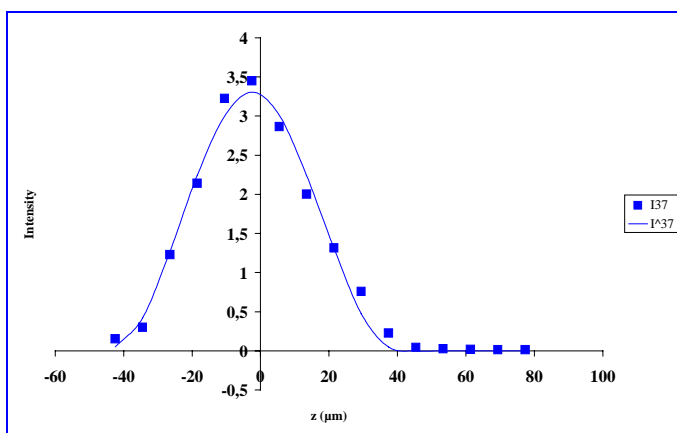


Figure 2

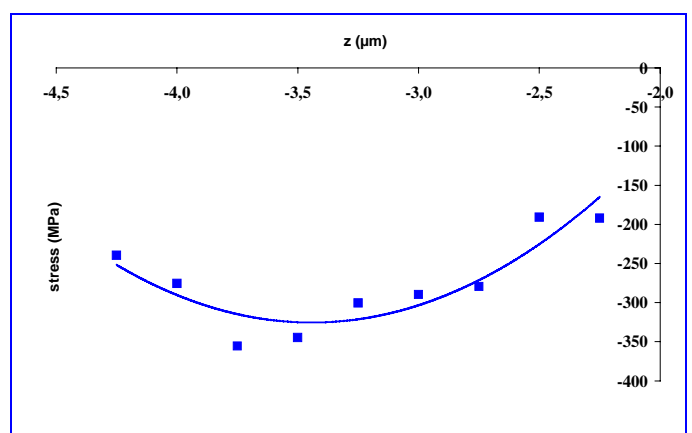


Figure 3

Stress results obtain for the superficial layers are shown in Figure 3.