ESRF	**Experiment title:** Durability of oxidized metallic materials studied by Laue micro diffraction	**Experiment number**: 32-02-736
Beamline: IF-BM32 GM CRG	**Date of experiment**: from: 8/09/2011 to: 11/09/2011	**Date of report**: 31/12/2013
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Report:

In oxidizing environments, the protection of metals and alloys at high temperature is provided by the growing oxide film. The efficiency of this protection can only be obtained if the formed film is well adherent to the metal (substrate) i.e. without microcracks and spalls induced by mechanical stresses. X-ray microdiffraction experiments are proposed to perform in situ local stress measurements in chromia thermally grown oxide (TGO).The main expected results are stress maps for three typical delamination figures. It will permit to precisely study the elasto-plastic behavior of chromia oxide thin films thermally grown on NiCr and FeCr alloys, from cracks initiation to thin film delamination and final spalling.

Feasibility tests on commercial chromia powders (1 μm in size) have been done using polychromatic micro beam XRD (see figure 1). It indicates that Laue patterns may be measured in growing oxide using white beam since chromia oxide grain size are expected to be in the range [0.1; 1 μm]. Unfortunately, the quality of the signal was not good enough for extracting valuable information concerning grain orientation and strains. Figure 2 shows a Laue pattern where both contribution of the substrate

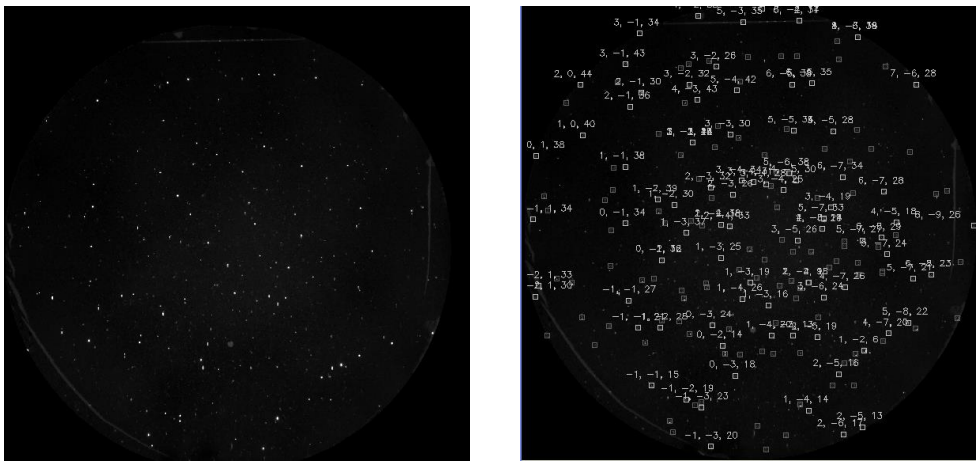

Fig. 1 Laue patterns after background subtraction and indexation (90 peaks)

(intense Laue spots) and the oxide scale (weak Laue spots) are visible. The XMAS software used does not allow indexing the diffraction from the chromia coating. The coherently diffracting domains are in fact smaller than the macroscopic grain size (optical image) and thus not really adapted to the beam size. In fact, the white beam which size is about 1.5 x 2 microns^2 illuminates then a large number of grains giving rise to complex Laue pattern. However, the substrate can be easily analyzed. This allows accessing the grain size distribution in the substrate below the delaminated area.

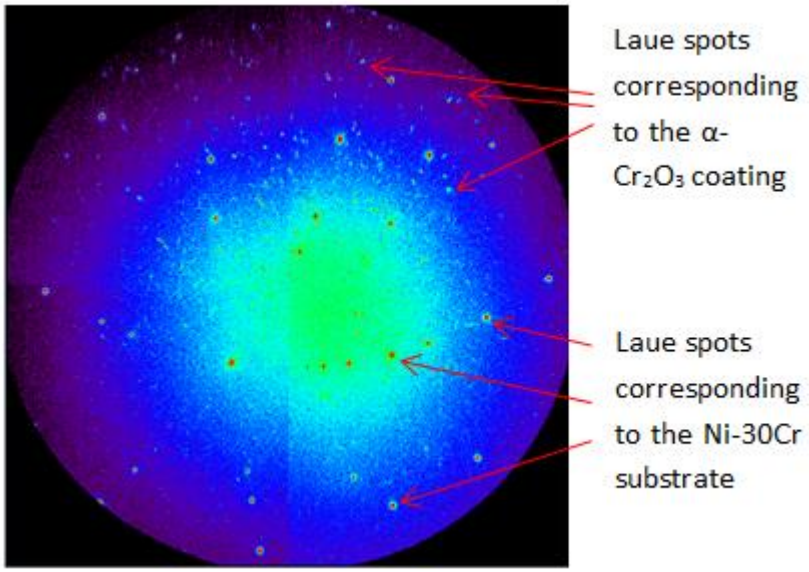

Fig. 2 Laue Pattern of Ni-30Cr oxidized during 18 h at 1000°C

It can be seen in figure 3 that buckling size may include several grains of the underlying substrate when the grain size is about 20-50 microns (Ni-Cr substrates). Delamination may also appear above a single grain when the grain size is about 100 to 200 microns (Fe-Cr substrates).

These first results show that grain size and crystallographic orientation of grains are not driving parameters for the localization of delamination process. Otherwise, residual stress level in metallic substrate can also be mapped. Whatever the considered point on the map, the stress values are close to zero. This result is in agreement with the equation of mechanical equilibrium of stresses, the thickness of the substrate (> 1 mm) being in the present case higher than the oxide coating thickness (< 10 µm).

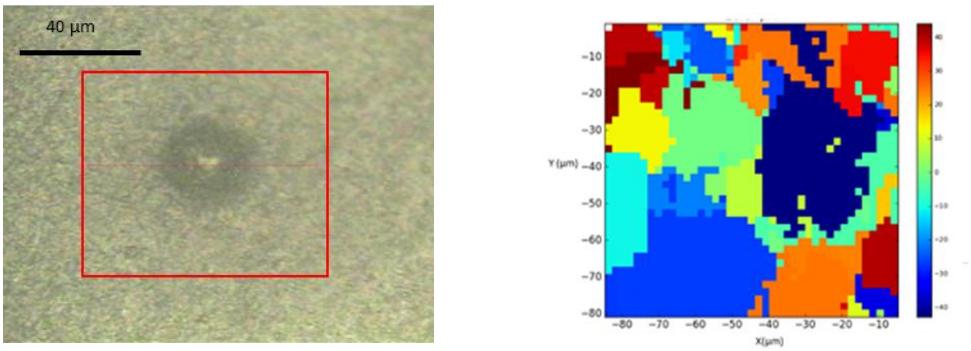

Fig. 3 (left) optical image of a buckling, (right) underlying grain orientation map (red rectangle) of Ni-30Cr substrate after 3h oxidation at 900°C

Complementary measurements have been done at ALS Berkeley using monochromatic microbeam, 10 x 10 micron^2 in size, in order to get stresses at the buckling scale. The measurements were successful despite the loss of spatial resolution for stress mapping. This explains the delayed report for our experiment at ESRF. The BM32 microdiffraction beamline is in fact well adapted for stress mapping measurements thanks to the online optical microscope; it allows a very precise observation of the buckling. Combined with fluorescence maps on markers, we are then able to precisely calibrate the X-Y stage for accurate positioning of the x-ray beam which is essential for mappings.

Publication(s):

- « Contribution to the study of stress release mechanism in chromia-formers materials : multi-scale approch by Raman spectroscopy and synchrotron diffraction », M. Guerain, PhD thesis, 5th of october 2012, Université de La Rochelle, France (in french).
- "Local stress determination in chromia formers : a way to investigate spontaneous delamination process" M. Guerain, P. Goudeau, J.L. Grosseau-Poussard, Journal of Applied Physics, 113 (2013) 063502
- "Micro XRD diffraction mapping of thermally grown Cr_2O_3 coating on NiCr/FeCr substrates: probing damages (the strain) at local scale", M. Guerain, G. Geandier, J.-S. Micha, C. Dejoie, N. Tamura, M. Kunz, J.-L. Grosseau-Poussard, P. Goudeau, in preparation.