

**(HT)-Raman microscopy to study (U,Pu)O₂ fuel
microstructure illustration on (U,La)O₂ and CeO₂;
first results on (U,Pu)O₂**

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[07.05]

Investigation on (U,Pu)O₂ fuel microstructure by Raman microscopy

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With the expansion of Sodium-cooled Fast Reactors, advanced new uranium-plutonium mixed oxide fuels (U,Pu)O_{2-x} with 19 < Pu/(U+Pu) < 30 mol.%, are currently developed. Their physico-chemical properties such as chemical homogeneity, oxygen stoichiometry (O/M ratio with M=(U+Pu)) and crystallographic structure, have to meet imposed criteria. As shown by recent studies, the Raman microscopy can be used to evaluate these properties at the grain scale. Moreover, with an adapted set-up, *in situ* measurements at high temperatures and controlled atmosphere can be performed.

Firstly, we have evaluated the efficiency of this approach on (U,La)O₂ and CeO₂ samples. These materials exhibit the same fluorite structure and share similar crystallographic properties with (U,Pu)O_{2-x}. A Raman imaging collected on an U_{0.75}La_{0.25}O₂ sample is shown in Figure 1. Both the intensity map of the T_{2g} line characteristic of the fluorite structure and SEM picture are presented. The microstructure of the sample can clearly be observed by the Raman imaging. Furthermore, even if both the XRD and the XAS results give an average O/M ratio of 2.00, other parameter maps revealed small local variations in both O/M ratio and La content.

Figure 2 shows the variation in the Raman line T_{2g} as a function of temperature on a CeO₂ sample during *in situ* measurements at high temperature under reducing atmosphere (pO₂~10⁻¹⁵ atm). The broadening and the shift to lower energies observed are due to both the thermal expansion and the reduction from CeO_{2.00} to CeO_{2-x}.

Finally, (U,Pu)O₂ samples with 19 mol.% Pu are being characterized at JRC Karlsruhe. Experiments are performed on sintered pellets and show variations in the T_{2g} line position as a function of the local plutonium concentration. Raman mappings of such samples are planned in the near future, as a new experimental set-up dedicated to fresh to (U,Pu)O₂ is now available at ATALANTE facility.

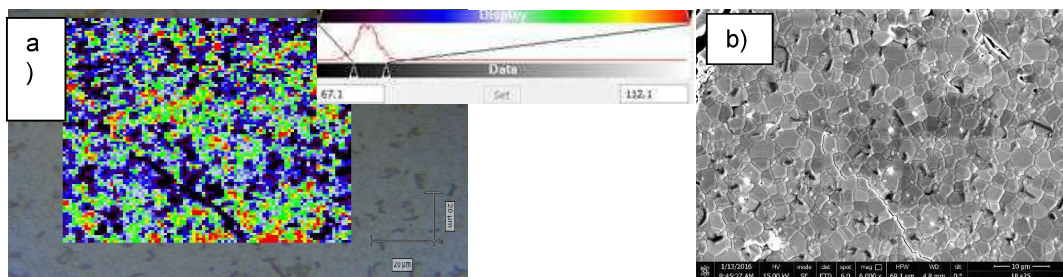


Figure 1: a) Map of the Raman T_{2g} intensity line (fake colors) obtained on U_{0.75}La_{0.25}O_{2-x} (100x100 μm), wavelength : 514 nm, 5mW, objective: x50, by InVia Reflex Renishaw b) SEM picture of the U_{0.75}La_{0.25}O_{2-x} sintered pellet

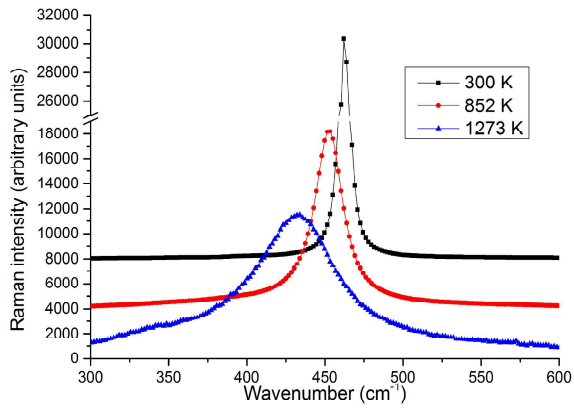


Figure 2: Variation in the Raman spectra (T_{2g} line) of CeO_2 with temperature in Ar

Keywords: Raman microscopy, (U,Pu)O₂ fuel, Crystallographic properties, Raman imaging