The experiment "MA 3618" was performed on the ID11 beamline from October 25th to 27th 2017

Fraction volume determination and lattice parameter evolution along the carburization depth of three different candidate steel grades for cladding

Background :

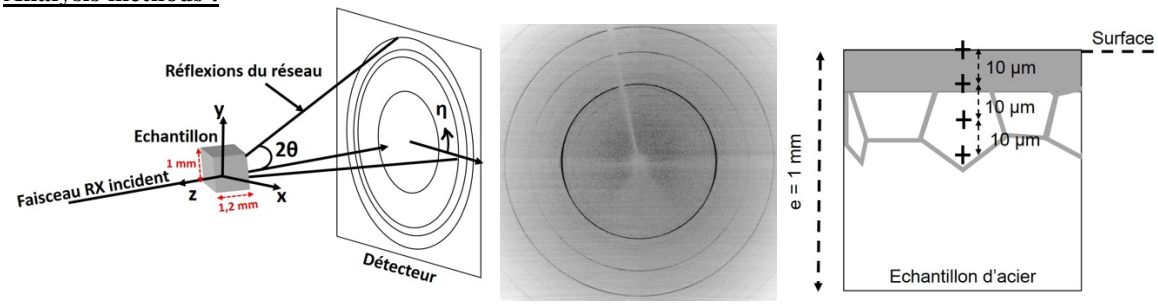
Study of the carburization of stainless steels in sodium medium for the development of the ASTRID fast neutron reactor prototype. More precisely the study of the corrosion behavior in the presence of sodium and B4C of steels constituting the control rods. According to feedback from old reactors, the lifetime of the cladding is limited by the kinetics of carburization of the cladding by B4C. Today the lifetime of the cladding is predicted using a conservative embrittlement law. In order to be less conservative, it is necessary to better describe the state of carburization of the steels under reactor operating conditions. Thus, in addition to carburization kinetics, crucial information on the nature of the carbides, their morphology, their location and their proportion in the steel is required. In order to analyze these quantities in detail, X-ray diffraction analyses of the synchrotron radiation were carried out through the section of sodium carburized samples. Below is the list of samples measured during the MA 3618 experiment.

Temperature	Exposure time	Steel	Maximum depth of carbon profile (EPMA)	Number
600°C	500 h	EM10, AIM1, 316L	EM10 (400 µm) AIM1, 316L (300 µm)	3
600°C	5000 h	EM10, AIM1, 316L	EM10 (500 µm) AIM1, 316L (500 µm)	3
500°C	500 h	EM10, AIM1, 316L	EM10 (200 µm) AIM1, 316L (150 µm)	3
500°C	5000 h	EM10, AIM1, 316L	EM10 (> 300 µm) AIM1, 316L (> 200 µm)	3
600°C	3000 h	304 (e=100µm)	Totally carburized	1
600°C	2000 h	EM10, AIM1, 316L (e=100µm)	Totally carburized	3
/	/	Initial samples 304, 316, EM10, AIM1		4
			TOTAL	20

Measurement parameters used :

The experiments were carried out on the ID11 beamline.
- X-ray diffraction in transmission (mirror Kirkpatrick-Baez)
- Freulon 4M detector, 2048 x 2048, binning 1x1, pixel size = 50 µm
- E = 65.4 keV
- Sample-to-detector distance = 0.305 and 0.324 m
- Beam size 10 x 100 to 800 μm^2
- Reference CeO2
- Counting time 1 s for EM10 and 0.5 s for AIM1 and 316L due to matrix saturation.

Analysis methods :



Diffraction profiles were carried out in the thickness of the samples with a 10 µm pitch. The MAUD and TOPAS software will allow to identify the nature and evolution of carbides as a function of the depth of the sample. In addition, the MAUD software will allow to quantify the evolution of the volume fractions of carbides considering the texturing of the samples. These results will be compared to the volume fractions determined with the TOPAS software which cannot calculate the texture.
Three steel grades have been studied, two austenitic 316L (15Cr10Ni) and AIM1 (15Cr15Ni0.4Ti) and one ferrito-martensitic EM10 (9Cr1Mo). Thus, the evolution of the nature and volume fraction of the precipitates will be studied according to the nature of the steels.
Precipitation will also be studied as a function of temperature (500 to 650°C). All these results will be compared with thermodynamic and kinetic predictions of carbide precipitation in steels (ThermoCalc and Dictra software).

Results:

The processing of the results initially concerns the samples carburized in sodium fuel at 600°C. First of all a first quantitative approach will be carried out (nature of the carbides and evolution of the nature and height of the peaks as a function of depth). This work has been done with the three steel grades 316L, AIM1, EM10. The natures of the carbides formed and the quantification of their volume fraction (figure 1) will be compared with the predictions of thermodynamic calculations. For the sample carburized at 500 °C very few peaks are visible. Precipitation kinetics being a thermally active phenomenon, it is possible that a very small proportion of carbides had time to precipitate.

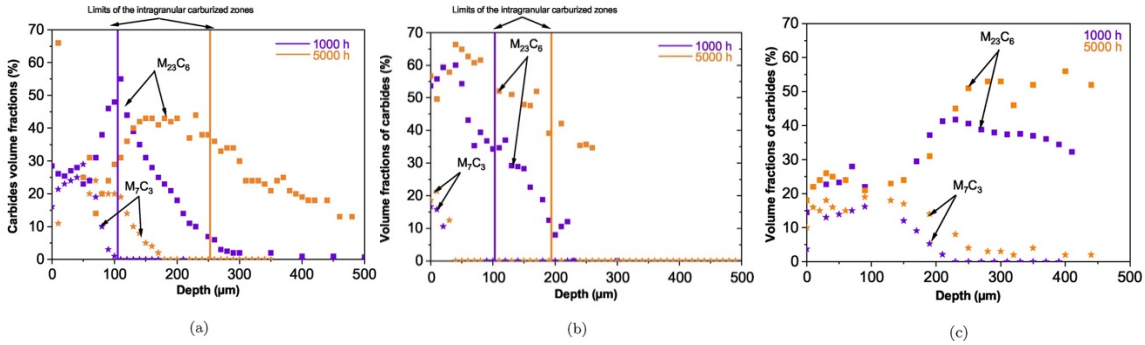


Figure 1: Carbide volume fractions in (a) AIM1, (b) 316L and (c) EM10 steels after 1000 and 5000 h exposure at 600 °C and $a_C > 1$.

For austenitic steels, the diffractograms performed in the thickness of the carburized sample at 600°C for 1000 hours are shown in Figure 1a and b. Only one diffractogram out of three was plotted over 500 µm thickness. The carbide peaks are no longer visible beyond 240 µm depth corresponding to the intragranular and intergranular carburization front on the optical image (figure 2b). This confirms that the configuration of the test made it possible to analyze both the carbides present in the grains and those formed in the grain boundaries.

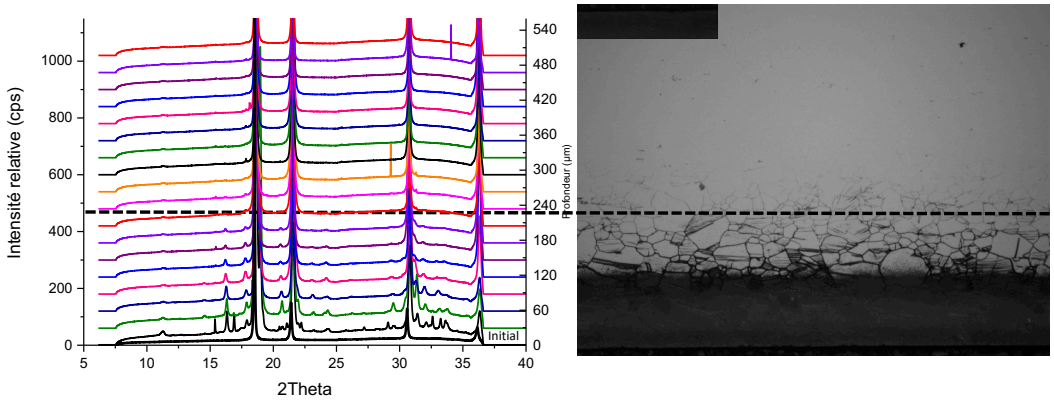


Figure 2: Left: Diffractograms made with a 30 µm pitch on carburized AIM1 steel 1000 h at 600 °C superimposed on the diffractogram of the initial uncarbonized sample. Right: Optical image of the revealed sample (ammonium persulfate 6V, 10s)

For more information please read the paper: **Corrosion Sciences 159 (2019) 108147**; Carburization of austenitic and ferritic stainless steels in liquid sodium: Comparison between experimental observations and simulations M. Romedenne, F. Rouillard, D. Hamon, B. Malard, D. Monceau