ESRF	Experiment title: XRF and XANES characterization of high p-doped SixGe1-x nanowires	Experiment number: MA-4642		
Beamline: ID16B	Date of experiment : from: 18/09/2020 to: 20/09/2020	Date of report : 21/01/2021		
Shifts: 9	Local contact(s): Jaime SEGURA-RUIZ	Received at ESRF:		
Names and affiliations of applicants (* indicates experimentalists): SOJO GORDILLO Jose Manuel; DUQUE SIERRA Carolina; MORATA Alex				

Report:

This proposal was partially allocated (only XRF experiments). During the 9 shifts, several SiGe NWs from 4 differente samples were dispersed on Kapton foils from 4 samples and characterized by XRF in order to elucidate the mechanism behind the Au-incorporation into the NWs during their growth, and the best growth conditions to avoid this effect. Table 1 summarizes the samples characterized during this experiment and their main characteristics.

Samples	SiH ₄ :B ₂ H ₆ ratio	Composition	HCl:Si ratio	Au inclusion in NW lattice
S1 (G670)	550	Si _{0.63} Ge _{0.37}	1.75	No
S2 (G665)	470	Si _{0.59} Ge _{0.40}	1.75	Surface
S3 (G673)	410	Si _{0.68} Ge _{0.31}	1.75	No
S4 (G677)	365	Si _{0.60} Ge _{0.30}	1.75	Yes

Table 1. List of samples proposed to be measured. All NWs are growth under the same conditions except for the SiH4/B2H6 ratio. The other growth conditions are: 180 min of growth time at 650 °C and 2.5 torr. Si:Ge ratio of 25.

Two representative XRF maps are displayed in the Figure 1. It can be observed that the Nws grown with low diborane concentration do not show a measurableAu concentration aside of the NP at the tip(used as catalyst of the growth). On the other hand, high diborane concentrations during growth conditions lead to the progresive inclusion of gold along the NW.





Figure 2. XRF maps of two NWs showing the Ge and Au distribution along the entire nanostructure. The pattern observed in the sample 1 (top) compared with the flat distribution observed in sample 4 (bottom) is considered to be produced by the high content of diborane in the growth process.



Figure 2. Calculated concentration profiles extracted from the XRF maps. Sample 1(top) clearly shows the absence of Au except at the tip, where the catalyst Au NP is located, whereas sample 4 (bottom) shows a notorious increase in the Au concentration along the NW. Right chart shows the summary of all concentration results of the study.

Figure 2 shows the longitudinal concentration profiles obtained for 2 representative NW studied (samples 1 and 4). The concentration trends can be observed in the rightmost chart. Results from the previous in-house measurements were included as well (HCl:Si ratios of 1.5) in order to further asses the effect of HCl as an effective agent for Au inclusion suppresion. However, Sample 2 measurements were not yet included in the study because later SEM analysis showed superifical NP. Beamtime is still necessary to perform the initially proposed nano-XRD and nano-XANES experiments to further asses whether this detected gold is superficial or included into the NW lattice. The later would modify the electronic structure of the crystal. Moreover, the HCl effect will be further studied using samples with higer HCl:Si ratios.