ESRF	**Experiment title:** Strain imaging in suspended GeSn micro-disks for laser application using Bragg ptychography	**Experiment number**: MA-3571
Beamline: ID01	**Date of experiment**: from: 8/11/2018 to: 14/11/2018	**Date of report**: 4/04/2018
Shifts: 18	**Local contact(s)**: Steven LEAKE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Vincent FAVRE-NICOLIN (ESRF)*

Joel EYMERY (CEA)*

Matthieu BERTRAND (CEA)*

Jeremy CHRETIEN (CEA)*

Gaetan GIRARD (ESRF)*

Samuel TARDIF (CEA)*

Vincent CALVO (CEA)

Vincent REBOUD (CEA)

Alban GASSENQ (CEA)

Report:

Objective

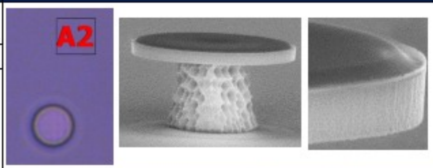
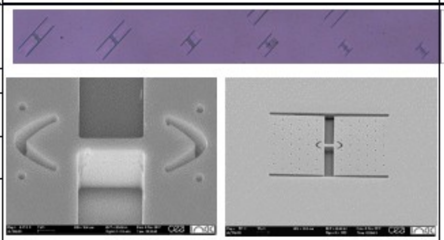
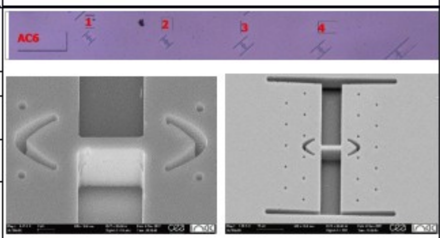
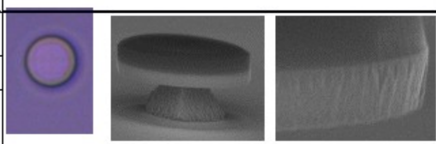
The microelectronics industry is actively looking for a CMOS-compatible material with a direct bandgap that could be used to fabricate an efficient monolithic laser source integrated on a Si platform, as alternative to the transfer of III-V based structures. $Ge_{1-x}Sn_x$ has been identified as a novel, viable candidate, with properties somewhere between its pure components : the direct bandgap of Ge is only slightly higher than its direct bandgap, while the direct bandgap of %alpha-Sn is negative.

While so far the laser regime has only observed at cryogenic temperatures in GeSn structures [1,2], strain engineering enables modifiyng materials' properties and thus raising the operating temperature. The initial epitaxial growth of $Ge_{1-x}Sn_x$ layer on Ge substrates usually results in a detrimental compressive strain in the layer. Indeed, larger Sn concentrations are required to achieve a direct bandgap if the layer is compressed : a direct bandgap $Ge_{1-x}Sn_x$ layer under -0.5% compressive strain (relaxed) requires x > 0.11 (0.08). Development of etching recipes selective against Ge and GeSn has allowed to consider suspended structures, that can relax the epitaxial strain. Laser effect with lower Sn content were thus recently achieved in suspended $Ge_{0.915}Sn_{0.085}$ micro-disks [3], and in micro-disks made by the co-applicants at the CEA.

The data collected during the beamtime aim to qualify the relationship between the actual anisotropic strain distribution in the micro-disk and its optical properties.

Samples

We studied four different samples, described in the table below. Each of them consists in patterns of several microstructures, repeated with different characteristic parameters such as stacking of layers (with step-graded GeSn for instance), lengths or Sn content. Two are composed of micro-disks amd the two other contain micro-bridges.

Sample name ESRF	*Stack T -> B*	*Dimension T -> B (nm)*	*Structure*	*Parameter*	*Dimension (µm)*	*Images (optic and SEM)*	*ESRF Measurement*	*Comments*
GeSn3-D16S1043P23	GeSn	480	u-disk	Disk diameter	4,6,8,10,12,14		8µm disk A2	Bilayer GeSn ~120nm @ 11% ~ transit layer ~225nm @ 13% ; Etching without O2; Change between post and preview
	Ge	2500		Underetch	3 um			
	Si	Substrate	See appendix A					
GeSn6-1000G40-2000	GeSn	480	u-bridge	Arm length	50, 100:100:1400		AC3-22 AC3-23 AC3-25 AC3-26	Bilayer GeSn : ~240nm @ 11% ~240nm @ 14
	Ge	2500		Underetch	~8			
	Si	40		Bridge length	8			
	SiO2	2000		Bridge width	1.5			
	Si	Substrate	See appendix B & C					
GeSn6-1000G25-400	GeSn	480	u-bridge	Arm length	50, 100:100:1400		AC6-2 AC6-3	Bilayer GeSn : ~240nm @ 11% ~240nm @ 14%
	Ge	2500		Underetch	~8			
	Si	25		Bridge length	8			
	SiO2	400		Bridge width	1.5			
	Si	Substrate	See appendix B & C					
GeSn4-D16S1044P11	GeSn 16%	240	u-disk	Disk diameter	4,6,8,10,12,14		8µm disk J1	Graded layer of GeSn
	GeSn 12%	120		Underetch	3 um			
	GeSn 10%	120	See appendix A					
	GeSn 8%	120						
	Ge	2500						

Experimental method

We used a monochromatic, KB-focused beam of size 400x175 (HxV) getting a flux of few 10^9 ph/s at 8keV. A maxipix detector was used during the entire experiment, although a bigger detector would have been more helpful as we expected to see very broad diffraction pattern but unfortunatly the Eiger of ID01 was not working at that time. For each sample we first measured the incident X-ray beam using a standard Siemens star target, performing 2D transmission ptychography on spiral-scans, which gives us an accurate estimation of the beam profile and focus position, key features for phase retrieval of Bragg Ptychography.

For each sample we used a microscope to get clear location of objects of interest, and then removed it to reach Bragg reflection of GeSn.

For the first sample, we tried to perform the experiment relying on this mapping, but the micro-disks are really difficult to align blindly i.e. with only angle scans and rocking curves : a 8 microns-diameter disk upon a 3 microns-wide pillar is almost impossible to align perfectly with the axis of rotation. This, combined with the wide scattering due to the mushroom-shape, leads to a high inaccuracy on the position of the beam relative to the sample, compromising the convergence of reconstruction.

For the two samples composed of micro-bridges, we were able to used the K-Map technique of ID01 to get mappings of the target bridge, in particular using the scattering at Ge & GeSn (113) peak (figure 2). Plus we managed to install a fluorescence detector (via an APD) which was very helpful used at Sn edge to get a clear location of the target bridge's center, by orthogonal scans across and along the bridge (figure 1). A lot of efforts was put in measuring repeatedly (004) & (113) rocking curves on a maximum number of bridges in order to assess the distribution of strain.

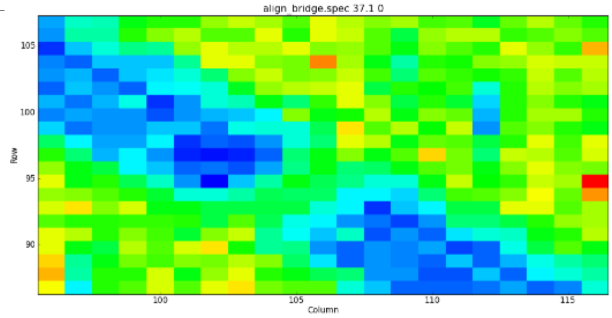


Figure 1 : Fluorescence map over a bridge

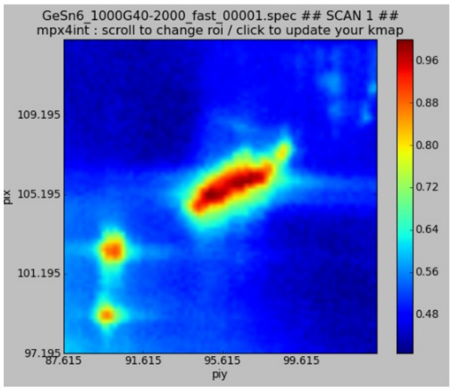


Figure 2 : K-Map around (113) GeSn peak

After collecting data from timescan to evaluate the beam damage, we were able to perform spiral-scans (400 pts, 1s/pt) at both (004) and (113) GeSn Bragg peaks at several eta angles (~12) , and at different positions on the bridges : center (where there is the largest longitudinal strain), upstream and downstream, the last two in order to get as close as possible from the frontier between the bridge and the arms which present the highest inhomogeneous strains. The techniques of 3D Bragg Ptychography and Back-Projection multi-angles Bragg ptychography will be used to invert these datasets, but until now the algorithms are not robust enough to converge with our data.

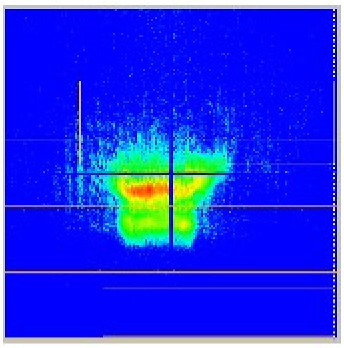


Figure 3 : Diffraction pattern of a (113) GeSn Bragg peak from a bilayer sample

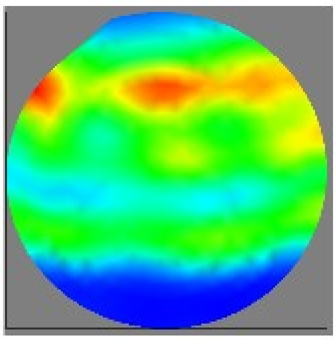


Figure 4 : diffraction at high eta-angle, 'disturbed' layer of GeSn

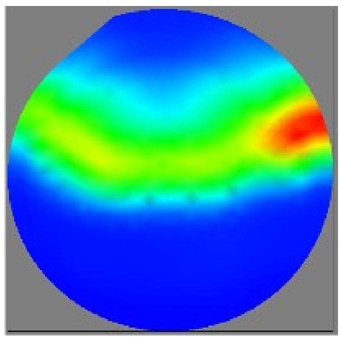


Figure 5 : diffraction at low eta-angle, 'homogeneous' layer of GeSn

Last but not least, we studied the micro-disks with step-graded GeSn stacks (last sample). The challenging part was to align the disk with the center of rotation, while avoiding the incident beam to impinge on the Ge pillar which leads to parasitic scattering. We managed to perform some spiral-scans at different location on the disk but the window of the detector might has been too small to catch the entire diffraction pattern. (figure 6,7)

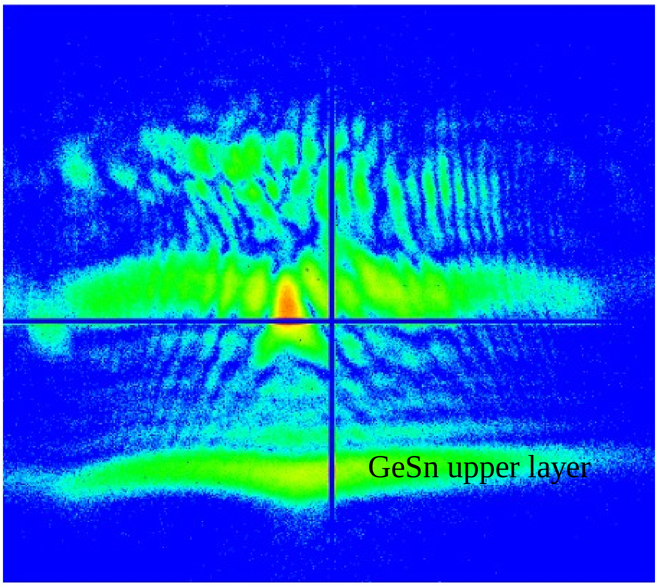


Figure 6 : Diffraction pattern of (004) GeSn Bragg peak, in which we can clearly see the different layers of the step-graded GeSn (ranging vertically), with differents level of complexity according to the purity of the layer : the upper GeSn layer is more relaxed and present less defects, thus its diffraction pattern is separated from the other layers (bottom).

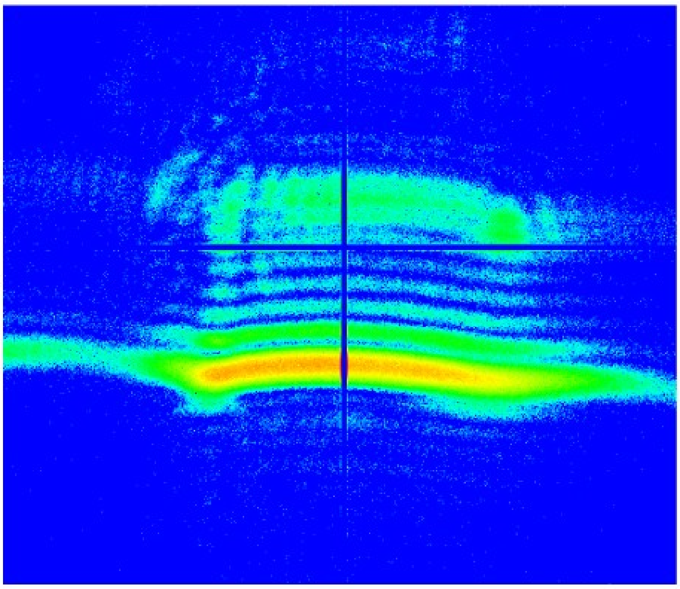


Figure 7 : Same as figure 6, at a different location on the disk

Conclusion

This experiment was very successful as we collected data for numerous analysis, including average strain analysis and more precise measurement as 3D and Back-Projection Ptychography. However we were unlucky with the detector being too small to cover the all range of diffraction from the micro-disks, and the alignement was made very complicated because of the wide tilt of the mushroom-shape object. However, we succeed to perfom promising spiral scans on micro-bridges and to demonstrate that the less distrubed layer of GeSn in a step-graded sample can be isolated and thus reconstruct if the quality of data is sufficient.

The algorithms for data analysis are still being perfected, notably for Back-Projection Ptychography which should lead to the most robust reconstructions, and we hope for an other round of measurements in order to get wider diffraction pattern with the EIGER detector, both for micro-disks and micro-bridges.

References

[1] S.Withs *et al.*, Nat. Photonics 9, 88 (2015)
[2] S. Al Kabi *et al.*, Appl. Phys. Lett. 109, 171105 (2016)
[3] D.Stange *et al.*, ACS Photonics, 2016, 3 (7)