Project title: **NAtuRal instability of semiConductors thIn SOlid films for sensing and photonic applications (NARCISO)**
Grant Agreement: 828890
Call identifier: H2020-FETOPEN-2018-2020/H2020-FETOPEN-2018-2019-2020-01
Start Date of Project: 01/03/2019
Duration: 42 months

Deliverable D2.1– V1.0

Title: Management Guidelines: financial management and quality procedures

Leader partner	AMU
Author(s)	Marco Abbarchi, Monica Bollani
Estimated delivery	30/11/2020
Actual delivery	
Revised version	
Work Package	WP2- Spontaneous spinodal dewetting
Tasks	2.2
Dissemination level	Public
Version	1.0

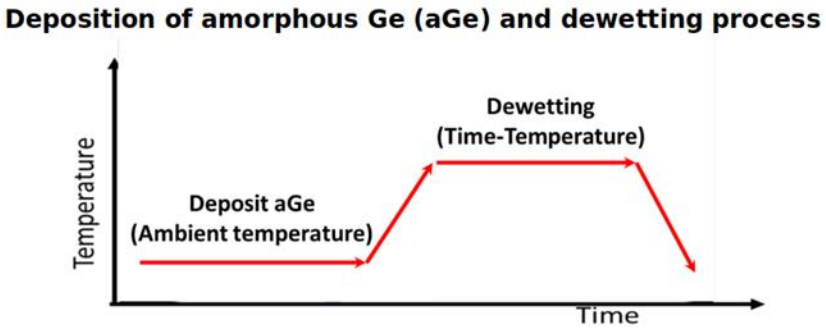
Contents

1. Introduction

We report on the achievement of solid state dewetting on 2 inches wafers of amorphous germanium on SiO2. This process has been completely customized in the AMU clean room and avoids the use of commercial products (e.g. avoids the use of commercial and expensive silicon (or germanium) on insulator substrates (SOI or GOI) that are provided in a limited set of specifications). The CNR also received the material as grown from AMU and reconfirmed the dewetting process on amorphous germanium using annealing systems present in the LNESS laboratory.

2. Dewetting process description

The full process starts with the fabrication of a thermal oxide 150 nm thick atop a 2 inches, bulk Si wafer. This is done by annealing at high temperature in a rapid thermal annealing oven (RTO, 950 °C-120min). The use of the RTO allows us to set an arbitrary thickness of the SiO_2, unlike with commercial SOI and GOI. The samples are then introduced in a molecular beam epitaxy (MBE) reactor in ultra-high vacuum (~10-10 torr). At room temperature, a thin Ge layer film is deposited (thickness between a few nm up to 200 nm) atop SiO_2. In this condition the Ge layer is amorphous. In this case we avoid any chemical cleaning (no flash) and we proceed directly to the dewetting step. The process is illustrated in Figure 1:



Reference sample (06/02/ 2020)	Starting structure	*Ex-situ* cleaning	Flash(time-Temperature)	growth before dewetting	Dewetting (time-Temperature)
MHM120-25	SiO2= 150 nm/Si	Without chemical cleaning	Without flash	aGe 5 nm	30'-780°C
MHM120-26	SiO2= 150 nm/Si	Without chemical cleaning	Without flash	aGe 10 nm	30'-780°C
MHM120-27	SiO2= 150 nm/Si	Without chemical cleaning	Without flash	aGe 15 nm	30'-780°C
MHM120-28	SiO2= 150 nm/Si	Without chemical cleaning	Without flash	aGe 30 nm	30'-780°C

Figure 1 Top panel: scheme of the dewetting process in the MBE. Bottom panel: description of the main features of 4 samples of aGe deposited atop the SiO_2 and dewetted in situ.

After the optimization of the growth and dewetting processes, we produced 4 samples by increasing the aGe thickness: 5, 10, 15 and 30 nm and keeping all the others parameters unchanged (dewetting temperature 780 C and time 30 minutes for all the samples).
All these samples have been obtained on 2 inches wafers. The amorphous layer on top is confirmed by RHEED analyzed: not diffraction points are observed, corresponding to the amorphous film.

Figure 2 RHEED map of initial amorphous film.

3. Samples characterizations

Figure 3 shows the SEM characterization for two different 2 inches wafers, for 10 and 15 nm thickness.

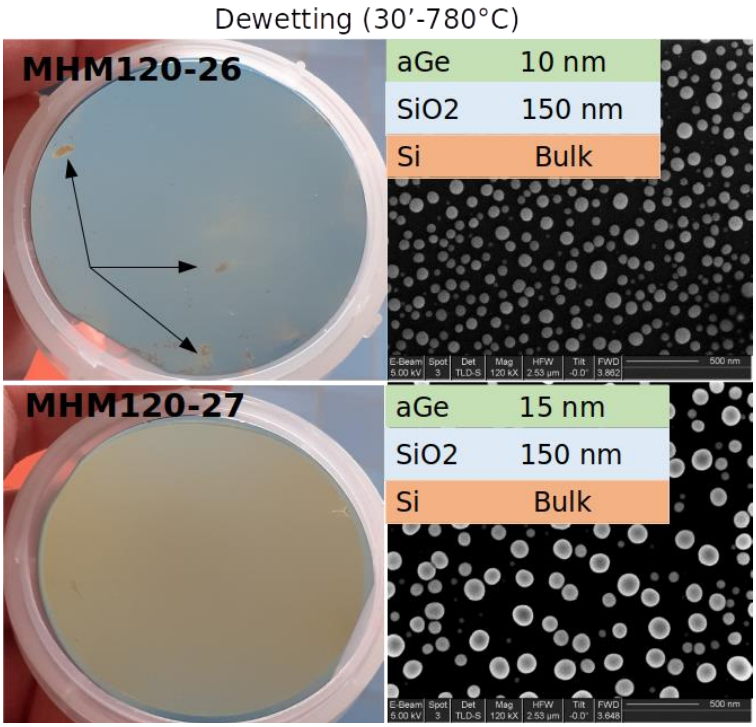


Figure 3 Left panels: 10 (top) and 15 nm (bottom) aGe deposited on 150 nm SiO_2 and dewetted at high temperature. On the right panels the corresponding scanning electron microscope micrographs (SEM) are reported

At this temperature and dewetting time the aGe layers for all the thickness investigated is completely dewetted. Generally speaking, we observe that for aGe thickness below 10 nm the process is not perfectly homogeneous (as highlighted by the arrows in the top left panel in Figure 3) whereas for larger thickness the quality of the dewetting is improved (e.g. the colorization is perfectly homogeneous and no defects are found on the sample surface). We also note that in these annealing conditions, the layer is completely broken in separate islands featuring a quite circular shape. This is typical of the equilibrium shape of crystalline germanium and is the signature of an advanced stage of dewetting. The same features are observed on all the dewetted samples as shown in Figure 4:

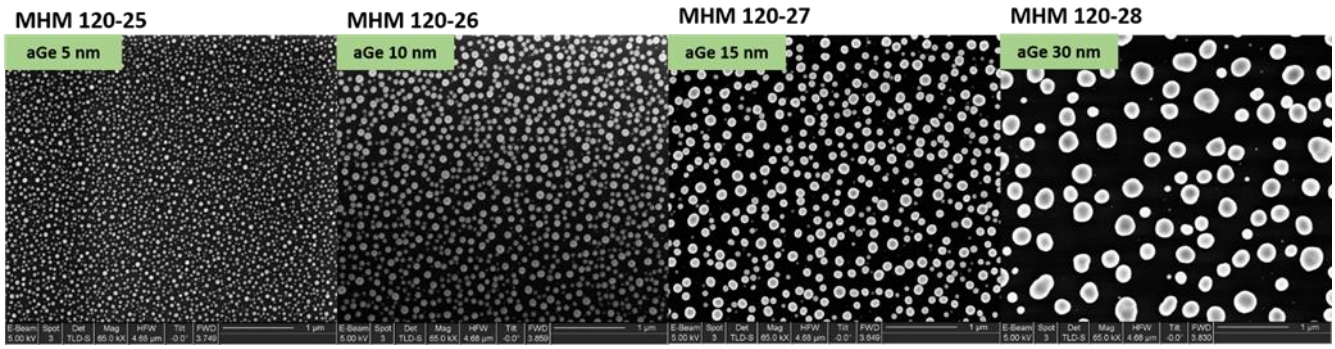


Figure 4 Left panels: sample reference (from top to bottom: 5, 10, 15 and 30 nm aGe after dewetting). Right panels: SEM images from each sample.

In analogy with the case of mono-crystalline SiGe deposited on SOI (Salvalaglio et al., PHYSICAL REVIEW LETTERS 125, 126101 (2020)) the final dewetting morphology can be tuned at will to obtain a spinodal pattern. Here, for instance, we show the cases of 50 nm thick aGe for two different annealing temperature in Figure 5:

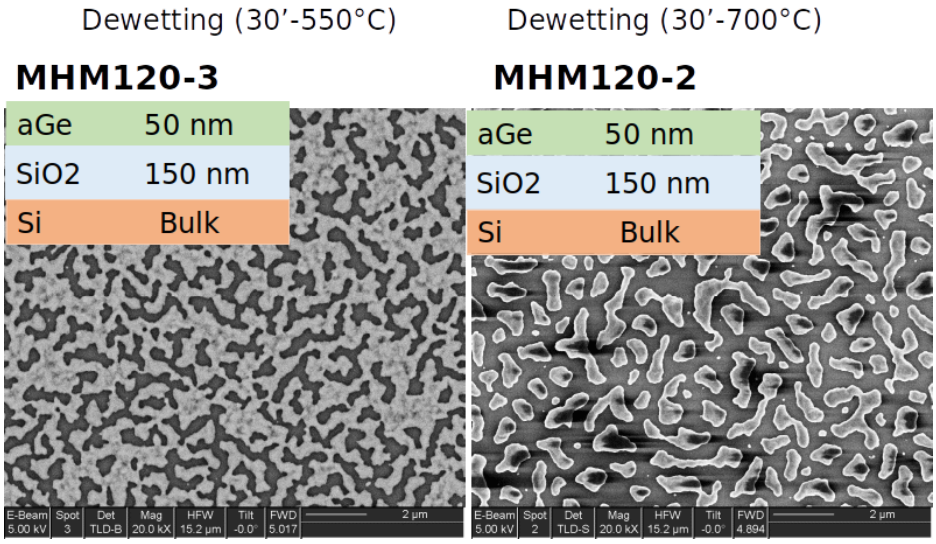


Figure 5: Examples of spinodal like dewetting of 50 nm thick aGe on SiO2.

4. Transfer of 2 inches dewetted wafers by nano-imprinting lithography

As final example of dewetting on 2 inches we show the case of islands transfer in a polymer matrix (see A. Benali et al 2020 J. Phys. Photonics 2 015002). Although via solid state dewetting it is possible to create many different kinds of ordered and disordered structures, a possible uses are limited by the presence of a rigid, bulky and non-transparent silicon substrate. A full exploitation of our method for (e.g. in nano-photonics) requires further processing to release the islands from their original support (allowing for instance, their use for light transmission). A description of the method is described in Figure 6:

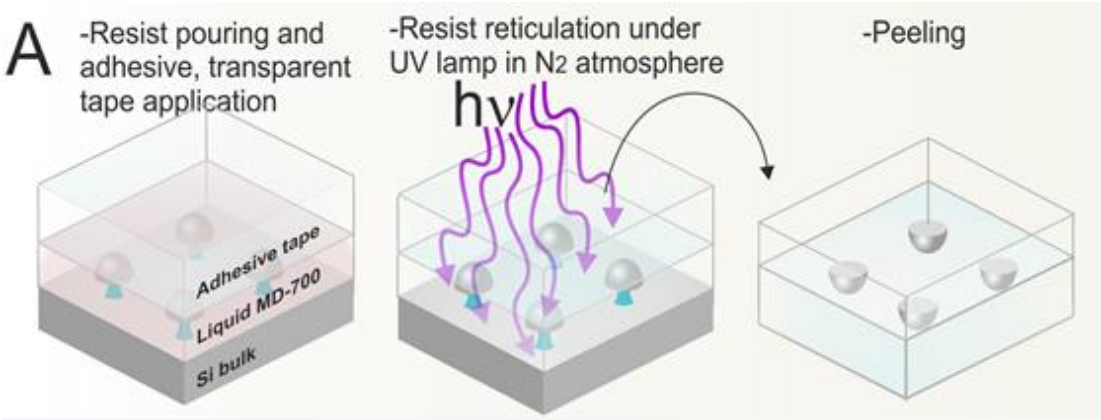


Figure 6: Islands transfer in transparent slices. After the annealing step, the SiO_2 underneath the Ge islands is partially etched in HF solution (not shown). A liquid UV-curable polymer (MD-700) is poured atop the islands and covered with a transparent adhesive tape. It is then cross-linked under near-UV light in N2 atmosphere for 30 min. The embedded islands are then peeled off from the original substrate. See reference A. Benali et al 2020 J. Phys. Photonics 2 015002

The procedure used for transferring the dewetted islands in transparent supports (figure 6) involves an etching step (not shown) for partially removing the SiO_2 by dipping the samples in a solution of HF for 10–30 min and then rinsing the sample by de-ionized water. This method is based on MD-700 (from Solway) UV-curable polymer. By pouring it on the dewetted islands after HF attack and covering with a transparent and adhesive plastic foil (Adwill D series UV-curable dicing tape from Lintec), the polymer is rigidified placing the ensemble for 30 min in N 2 atmosphere and exposing it to a LED emitting at ~365 nm (from Spectroline). Finally, the encapsulated islands are peeled-off.

To illustrate the transfer process, we consider a custom GOI. It is obtained by (1) processing a 2 inches Si wafer by RTO, forming a 130 nm thick thermal oxide, (2) Ge deposition (10 and 30 nm) by MBE and finally, (3) annealing at high temperature (as described in figure 1). This sample is diced in 6 parts to study the effect of HF etching on the efficiency of the transfer process. We perform a systematic investigation of the efficiency of the transfer process for 10–50 min HF etching assessing the yield of the transfer process monitoring the number of un-transferred particles left on the original substrate (not shown). We observe a decreasing trend of the number of un-transferred islands up to a minimum of ~4% for 30’ etching whereas longer etching time provides

a much less performing transfer. In a different case, for a 10 nm thick aGe layer and 25' HF etching, we were able to obtain even better results, with a transfer yield as large as 99.94%.
By monitoring the light diffusion we observe the presence of size-dependent resonances with a peak at about 900 nm for the larger particles obtained from 30nm aGe dewetting, and at 700nm for the smaller ones from 10nm aGe (figure 7 b, top and bottom panels respectively). These bands are interpreted as multi-polar Mie modes sustained by the particles, as expected for this kind of devices.

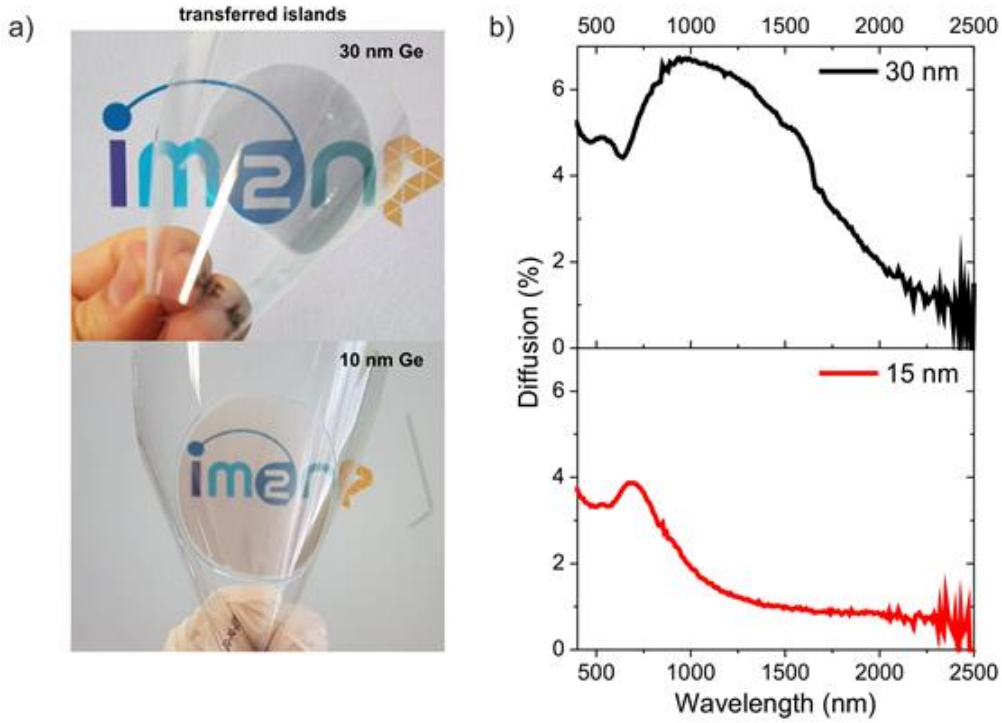


Figure 7"Light scattering from plastic-embedded Ge islands" (a) Ge islands obtained dewetting a 30nm (10 nm) UT-GOI embedded in plastic (b) Top (bottom) panel: reflected diffusion from 30nm (10 nm) dewetted UT-GOI., See reference A. Benali et al 2020 J. Phys. Photonics 2 015002

By monitoring the light diffusion we observe the presence of size-dependent resonances with a peak at about 900 nm for the larger particles obtained from 30nm aGe dewetting, and at 700nm for the smaller ones from 10nm aGe (figure 7 b, top and bottom panels respectively). These bands are interpreted as multi-polar Mie modes sustained by the particles, as expected for this kind of devices.
Analytical simulations of the scattering cross section for spherical, Ge-based particles embedded in a n = 1.5 refractive index material (that is close to that one of the polymer embedding the Ge islands), are in qualitative agreement with the experimental scattering spectra (Figure 8 top panel): for 200 nm diameter, Ge-based spherical particles (close to the diameter measured for 30 nm UT-SGOI dewetting) we observe a scattering peak at about 900nm. Similar arguments hold for the particles formed from the 10nm thick GOI.

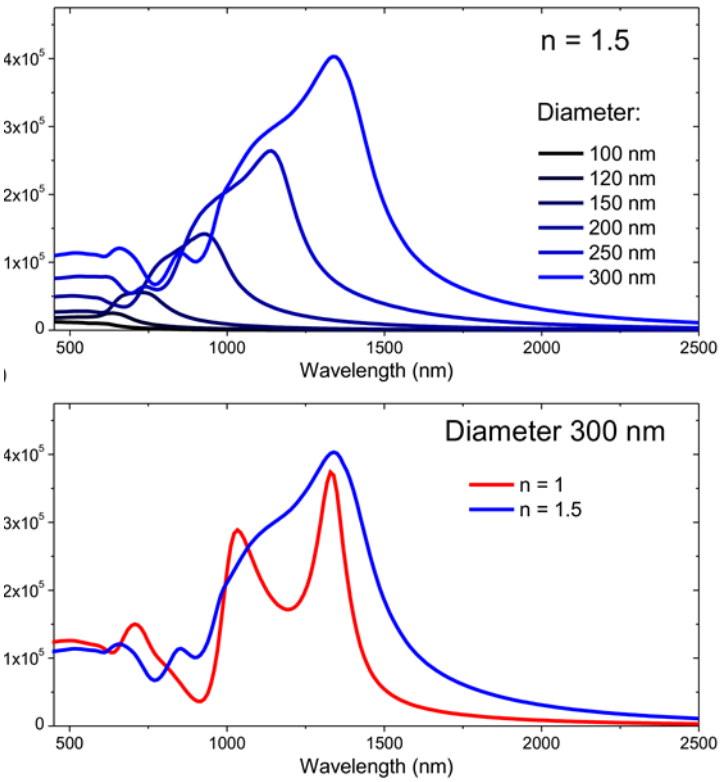


Figure 8 Analytical simulation of the scattering cross section for Ge-based spherical particles with variable size, embedded in a n = 1.5 refractive index material. (d) Analytical simulation of the scattering cross section for Ge-based spherical particles with diameter 300 nm in vacuum (red curve, n=1) and embedded in polymer (blue curve, n=1.5).