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PROMISE

PROtotypes of Magnetic Imaging Systems for Europe

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= Deliverable D1.1 =

User requirements

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Executive Summary

This document lists all material requirements and experimental conditions to ensure measurements and enable the successful execution of the use cases. It is the first technical step to reach TRL7 at the end of PROMISE.

Each end user has proposed general conditions, needs, requirements and limitations to achieve meaningful measurements on their samples of interest. To this end, each end user will provide different samples with increasing complexity to match their final samples of interest. This means that samples will be designed with specific characteristics that will be modified up to industrial or research samples. GSEMI has outlined the requirements for graphene and graphene chips, which are crucial to execute the use case in T5.1. Airbus CRT has specified the aluminum alloy, coating system, sample geometry, preparation and experimental conditions for measurements in T5.2, ensuring comparability of results. MF has defined two samples of different geometry of its micromagnet array, both directly related to its interest, to be considered in case T5.3. UPV/EHU has defined magnetic nanostructure specifications for T5.3, focusing particularly on the vortex spin configuration for biomedical applications in T5.4. UPV/EHU has furthermore listed requirements not only to reveal the interaction between magnetic nanostructures and cells, but also the requirements to keep the cells alive and measure their bioavailability.

Sample specifications and measurement requirements have been discussed with each of the end users and with all developers participating in PROMISE.

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Table of Contents

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1. Introduction

Deliverable D1.1 summarizes the requirements for each prototype from the point of view of the needs and constraints of the use cases. D1.1 belongs to WP1 and together with D1.2 will set clear the development of the two prototypes in WP4 and will guide the requirements and constraints for a successful outcome in the measurement of the different use case samples in all tasks (T5.1 to T5.4) in WP5.

The details of this deliverable have been discussed partially or in its totality by the following participants:

Name	Surname	Organisation
Abdellatif	Bachar	MAGNETFAB
Hans	Bartling	TNO
René	Böttcher	AIRBUS
Lola	Boyano	UPV/EHU
Milan	Calic	MAGNETFAB
Estibaliz	Garrote	TEC
Johannes	Lang	DIA
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Amaia	Rebollo	GSEMI
Xavier	Vidal	TEC
Amaia	Zurutuza	GSEMI

2. Specifications

This report defines the requirements for materials, components and experimental conditions to ensure comparability of measurements and enable successful execution of the use cases. It focuses on the samples provided by the use case partners.

2.1. Graphene chips

Graphene chips are provided by GSEMI. Currently, the measurements will be compared with GSEMI's electrical quality control measurements, which gives average mobility of charge carriers information. These samples are related to T5.1.

These are the common features of all samples provided by GSEMI:

Sample details:

1) Graphene sheets are 20 µm thick or more
2) Roughness sub-nanometer

Goals:

1) Current flow
2) Enhance defect identification and current flow imaging in graphene
3) Temperature changes
4) Characterization of charge carrier mobility through the graphene sheet

General restrictions:

1) DC current < 100 µA better below 25 µA

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2) Laser power < 1 mW / μm^2
3) Attention to potential heat transfer from the graphene sheet to the NVs during the measurements

Type of measurements. In this case all measurements have to be done in DC operation:

1) ODMR
2) Magnetic sensitivity
3) Vectorial
4) Mapping of electrical current (DC)

To reach a successful process on commercial graphene chips three different samples will be prepared by GSEMI. The third is the configuration used commercially.

GSEMI Sample 1: **Graphene deposited on diamond on NV side**

- No specific conditions.

GSEMI Sample 2: **Graphene deposited on diamond on opposite NV side.** These samples are an option in case a charge transfer is observed in Sample 1.

- No specific conditions.

GSEMI Sample 3: **Graphene deposited on silicon**

- Specific restriction: Diamond should not touch graphene to avoid scratching.

2.2. Al alloys and conductive samples

Aluminium alloys and conductive samples are provided by AIRBUS. These samples are related to T5.2. The samples will be compared with data obtained by the scanning vibrating electrode technique (SVET), which is the main technique used by AIRBUS for characterization. This technique requires long duration measurements, and the sample position is 20 microns away.

These are the common features of all samples provided by AIRBUS:

Sample details:

1) Sheets of about 1 mm thick and adaptable geometry; standard size is (2 x 2) mm^2.

General restrictions:

1) Sensor, particularly the NV layer, has to be fine with a liquid containing salt.

Type of measurements:

1) Different distances between NV layer and Al alloy

AIRBUS will provide four different types of samples. Three of them belong to an evolution to reach the final target sample. Final sample that meets the characterization needs for corrosion detection on polymer coated Al alloys. The increased complexity of the samples is intended to help the prototypes to reach an optimal configuration and isolate possible barriers. The fourth sample will complement the wealth of results of interest by detecting cracks in conductive materials.

AIRBUS Sample 1: **Dummy sample "Intermetallic Particle"**. Artificial interface of Cu and Al (e.g. Al wire embedded in Cu block). Size depending on wire thickness (proposed: 1x1 mm^2).

- Goal: Estimate feasibility of corrosion current measurement and comparison with existing SVET data.
- Specific restrictions:

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a) Simultaneous measurement of the sample surface of an area about (1x1) mm^2, to gain a benefit compared to scanning methods (e.g. SVET).

b) Sample needs to be immersed in an aqueous electrolyte (min. 0.1 mol/L NaCl, max. 5 wt% NaCl). Otherwise, no corrosion currents will be present.

c) Experiments performed at ambient temperature, optional up to 50°C (external heating sufficient).

d) Lateral resolution must not exceed IMP size (1-20 µm)

e) Time resolution in range of seconds desirable

f) Optional: Inclusion of a counter electrode on the surface of the sensor (e.g. Pt layer, compact or mesh-like) required if sample should be polarized in order to force higher corrosion currents, requires parallel orientation of counter electrode and sample's region of interest (ROI) - allows combination with various electrochemical methods.

- Type of measurements:

a) Average local current density (Hot spot identification)

b) Current density transient (Hot spot evolution/corrosion progress).

AIRBUS Sample 2: **Al alloy AA2024**. Cu-rich Al-alloy with geometry: Flat rolled metal sheet (0.8-1.2 mm thick, 1x1 mm^2 up to 80x150 mm^2). Surface as is or treated if required (e.g. sanding, polishing, chemical activation). It will be considered natural surface roughness (ca. 5-10 µm), without passivation or coating. Alternative Al-alloys can be used as well (e.g. Zn-rich: AA7075, AA7050).

- Goal: Monitor corrosion currents at corrosion hotspots and their evolution over time.
- Specific restrictions: Same as sample 1.
- Type of measurements: Same as sample 1.

AIRBUS Sample 3: **Polymer-coated alloy AA2024**. Substrate identical to Sample 2, except additional TSA (anodized metal-surface). Inhibitor-free coating (20-60 µm thick, variable), PU- or Epoxy-based. Inclusion of a U-shaped defect (1 mm wide, ca. 200-300 µm deep).

- Goal: Monitor corrosion currents at corrosion hotspots focussing on the polymer-substrate interface at the sides of the U-shaped defect and their evolution over time (underpaint corrosion).
- Specific restrictions:

a) and c) to f) as Samples 1 and 2.

b) Sample defect volume needs to be immersed in an aqueous electrolyte (min. 0.1 mol/L NaCl, max. 5 wt% NaCl) - otherwise no corrosion currents.

- Type of measurements: Current density transient (corrosion progress).

AIRBUS Sample 4: **Conductive (metallic) sample with incorporated cracks**. Might be identical to Sample 2, but adjustable (e.g. Al-alloys, Ti-alloys, Steel etc.). Sample deformed (linear, bent etc.) in order to incorporate defect/cracks.

- Goal: Detect cracks in the material by measuring Eddy currents.
- Specific restrictions:

a) Sequential measurement acceptable but simultaneous measurement of sample surface desirable.

b) No additional media (e.g. electrolyte) required.

c) Experiments performed at ambient temperature.

d) Lateral/crack size resolution in the range of µm (Eddy current) to nm (post Eddy current).

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- Type of measurements:
    a) Detection of position of cracks (qualitatively, imaging).
    b) Detection of size of cracks (quantitatively).

2.3. Micromagnets

Arrays of micromagnets are provided by MAGNETFAB. These samples are related to T5.3. The aim is to reach a spatial resolution of the magnetic field below 15 µm, which is the current resolution obtained with CMOS camera.

These are the common features of the arrays provided by MAGNETFAB:

Sample details:

1) Si wafer containing thousands of integrated NdFeB micromagnets.
2) Different heights, but maximum thickness 50 microns.

Goal:

1) Sub-micron spatial resolution.
2) Integrated air-gap measurement (ensure all the measurements are done at the same distance). We might consider an approach by interferometry to obtain the distance between NV layer and micromagnet arrays.
3) Defects detection (e.g., cracks).

Type of restrictions:

1) Avoid any contact with the samples.
2) Attention to the ambiguity between signal strength and distance during measurements.
3) Wafer 200mm diameter, so wide space is required for sample holder
4) Measurement time for the whole wafer will be too long.
5) Strong signal that can induce spin-mixing on the NVs.

Type of measurements:

1) Magnetic field distribution
2) ODMR
3) Vector magnetometry
4) Fit with simulations

Two different sets of micromagnet arrays will be supplied where the goals, restrictions and required measurements are the same:

MAGNETFAB Sample 1: **Square 200 µm x 200 µm individual micromagnets in an array of 50 microns pitch**.

MAGNETFAB Sample 2: **Non-square shape individual micromagnet in an array of 50 microns pitch**. Same as sample 1

2.4. Microdisks and nanostructures

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Microdisks and nanostructures are provided by UPV/EHU. These samples are related to T5.3. Measurements will be compared with simulations. Moreover, some sample will be benchmarked with vibrating sample magnetometer and magnetic hysteresis loop.

These are the common features of all samples provided by UPV/EHU:

Sample details:

1) The samples are made of Ni80Fe20 coated with gold, but no coating and other coatings are also available.

Goal:

1) At some stage the dynamic response under alternating magnetic field in the frequency range between 1Hz to 20 Hz will be measured.

General restrictions:

1) Measurements need an external magnetic field of 5 mT, ideally up to 100 mT if saturation needs to be reached.

Type of measurements:

1) ODMR for stray field
2) Vector magnetometry
3) T1 relaxometry

Due to the richness in behaviour of the samples with respect to sizes, configurations and distribution, different sets of samples will be prepared by UPV/EHU. Five different sample configurations have been selected, also in coordination to the measurements that will be done on the cells.

UPV/EHU Sample 1: **Square array of magnetic microdisks on Si wafer**

- Goal: Detection of the magnetic vortex core and magnetic profile of the vortex state.
- Specific restrictions:
  a) Microstructures limited to the formation of spin-vortex configuration
  b) Attention to fluorescence from Si that can mask or add optical noise to the NV emission, it might be needed to measure in a different substrate as Ge

UPV/EHU Sample 2: **Square array of magnetic nanodisks on Si wafer.** Shape and size of nanostructures must host a spin-vortex state.

- Goal: Images of magnetic vortex states in disk-like nanostructures
- Specific restrictions: same as sample 1.

UPV/EHU Sample 3: **Pattern of elongated shape nanostructures (ESN) with aspect ratio 4:1 on Si wafer**

- Goal: Images of magnetic poles in the ESN with single magnetic domain.
- Specific restrictions: same as sample 1.

UPV/EHU Sample 4: **Nanodisks in suspension**

- Goal: Real-time detection of orientation and motion of nanodisks in suspension.
- Specific restrictions:
  a) Nanodisks might be dispersed far away from the sensor surface inducing a gradient of the magnetic field.
  b) The bias magnetic field can induce the particles to move accordingly.

UPV/EHU Sample 5: **Nanodisk directly on NV layer ordered and scattered**

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- Goal: Measure the movement of the nanodisks under an oscillatory magnetic field.

2.5. Skin cancer and healthy cells

Skin cancer cells and healthy cells as control are provided by the Faculty of Medicine (SM) of the UPV/EHU. These samples are related to T5.4. Two of the three different groups of samples are combined with the above-mentioned nanostructures, which are part of T5.3. The measurements will be compared with their current approach using SQUID and confocal microscopes for the viability of the internalization of the nanostructures by the different cell types. Attention should be paid to whether changes in cell counts and cell viability come from growth conditions on the diamond surface compared to the surface used for SQUID and confocal measurements.

These are the common features of all samples provided by the SM of UPV/EHU:

Sample details:

1) Cell size ranges from 10 μm to 60 μm, the most probable size being 20 μm.

Goal:

1) Temperature
2) Measure whether both cell viability and cell division occur as under normal conditions.
3) Detection of nanodisks inside cells or attached to their membrane.
4) Percentage of cells adhered to the nanodisks to control which is the most optimal adhesion surface for the cells.
5) Determine which physiological element or compound is optimal to track the cell dynamics; for instance, O2 concentration (hypoxy), PH, mitochondrial activity, ATP.
6) Determine parameters that allow differentiation of healthy cells with respect to tumoral cells.

General restrictions and considerations:

1) Functionalize the diamond to promote adhesion and facilitate comparisons with the usual surface used.
2) The cells need to be kept alive during transport.
3) Temperature of the chamber (or incubator) of the cells must keep constant conditions during transport and measurement time: 37 degrees, 5% CO_2 and not low humidity.
4) Potentially induced heating due to the excitation of the micro-disk at frequencies below 60 Hz, usually 10Hz.
5) Acquisition time: Due to the movement of the cells the acquisition time measurements must be below 1 hour for localization of the nanodisks with low spatial resolution and acquisition time measurements below 5 minutes for sharp images.
6) Volume and space to fit the chamber on the widefield prototype.
7) RF power or laser absorbed by the cells or the medium that might increase the temperature.

Type of measurements:

1) ODMR for stray field combined with optical image
2) Vector magnetometry combined with optical image
3) T1 relaxometry combined with optical image
4) Relaxometry for detecting a relevant physiological element to understand the state and type of the cell and the relation with the magnetic nanodisk.

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5) Long-time measurements for identifying movement of the cells and induced apoptosis of the tumoral cells.

Three different sets of samples will be prepared with the aim of obtaining different information of the cells while testing the widefield prototypes in different conditions.

SM of UPV/EHU sample 1: **Cells bound to nanodisks** prepared in current standard way and later transferred to the NV diamond substrate. Means healthy cells (melanocytes) and tumour cells (melanoma) internalize the nanodisks.

- Goal:
  a) Image the internalization of the nanodisks by the cells.
  b) Merge images of the optical microscope of the cells correlated with the magnetic signal of the nanodisk.
- Specific restrictions: Obtain a proper protocol for transferring the cells to the NV layer preserving a viability of 90%.

SM of UPV/EHU sample 2: **Cells bound to nanodisks directly on NV layer ordered and scattered**. Cells grown directly on the diamond substrate.

- Goal:
  a) Same as sample 1
  b) Same as sample 1
  c) Determine the diamond surface termination to not disturb both cell viability and cell division as in normal conditions.
- Specific restriction: Diamond termination might be important as the absence of good adhesion, cell viability decreases considerably.

SM of UPV/EHU sample 3: **Cells cultured without nanodisk.**

- Goal: Same as sample 2 c)
- Specific restriction: Same as sample 2.

3. Conclusions

Deliverable D1.1 will be crucial for the prototype and component developers not to neglect the needs of the end users. This is of utmost importance to obtain a prototype that after reaching TRL7 aspires to reach the market and to be useful as a versatile tool.

However, discussions between end users and developers have revealed many challenges in the treatment of samples that could be of difficult compatibility with NV technology. Therefore, some end-user samples are designed to successfully complete the measurement, where only but always the final sample is of industrial interest.

A complete list of samples, targets and requirements is given in D1.1, but they are not set in stone. Circumstances may change over time and may need to be adapted during development.

4. Degree of progress

This deliverable is a 100% complete. The specifications to define prototypes will be complemented by deliverable D1.2.

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