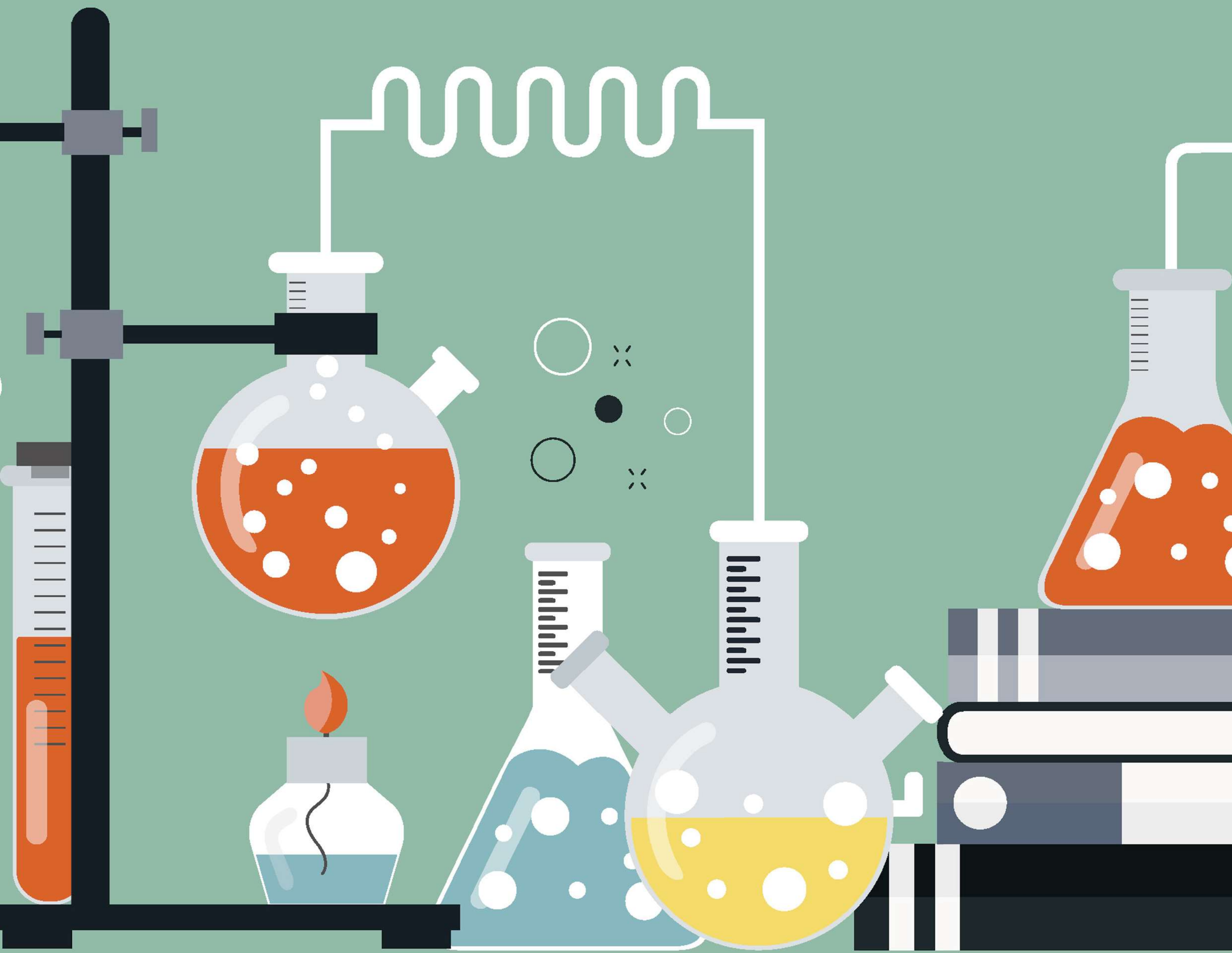
BEHIND THE SCENES

Nicolas Thenard, GTT, France, provides a detailed look inside the company's cryogenic materials laboratory.

To develop increasingly effective containment systems, knowledge of the performance of materials is essential. Since liquid gases are transported at cryogenic temperatures (-163°C for LNG), mastering and capitalising on material behaviour as a function of temperature are the main aims of GTT's materials department.

This department is divided into three sectors: metallic materials and welding; non-metallic materials; and R&D



materials. Each of these sectors has its own internal testing laboratory, based in Saint Rémy lès Chevreuse, France. The non-metallic materials department currently consists of 10 specialised engineers and five laboratory test technicians.

GTT has continuously invested in new cryogenic temperature measurement equipment, and the non-metallic materials laboratory has been renovated, allowing the installation of recent equipment acquisitions with optimum ergonomics. GTT has thus developed an improved tool, and can anticipate future industry requirements.

After several months of work, the non-metallic materials laboratory is fully operational once again. The new laboratory was officially inaugurated last May.

Figure 1. Photograph of the inauguration.

Figure 2. Tensile test specimen for cellular materials.

Figure 3. Guarded hot plate (GHP) image.

Focus on the non-metallic materials laboratory

The non-metallic materials laboratory now covers 700 m². It can perform practically all tests necessary to understand the performance of non-metallic materials used in already proven technologies, as well as those under development within the innovation division.

One of the advantages of this laboratory is that it gathers all of the test resources in a single centre of expertise, easily accessible to staff. It allows engineers to understand complex physical phenomena, maintain responsiveness through schedule optimisation, and ensure the confidentiality of results.

To be representative of the severe environment in which materials are used in GTT's technologies, all material tests must be carried out in a controlled atmosphere at cryogenic temperatures. This laboratory is therefore equipped with a liquid nitrogen network, connecting the test facilities directly to a 25 000 litre tank located near the laboratory. This tank operates at a pressure of 3.5 bar.

It is equipped with a remote sensing system to keep the liquid nitrogen supplier informed of the liquid level at all times and to trigger a delivery of liquid nitrogen as soon as it is necessary. GTT's non-metallic materials laboratory uses approximately 20 000 litres of liquid nitrogen per week. The network is divided into three independent vacuum lines to optimise pressure stability.

The inlet pressure for each test device is monitored to ensure that the tests run smoothly. The use of liquid nitrogen allows GTT to carry out all tests over a temperature range between -196°C and room temperature. The liquid nitrogen is then vaporised in the containment enclosures of each test device to adjust the test temperature.

Because of this high consumption, communal oxygen percentage detectors are placed at different locations in the laboratory, and connected to a central control station that can act on safety mechanisms. All staff are also equipped with individual detectors attached to their work clothes, in order to minimise any risk of anoxia. Personal protective equipment (PPE) is of course compulsory to avoid all other risks related to laboratory work.

Materials tests are carried out in accordance with international testing standards (ASTM, ISO, EN, DIN, etc.). Among other parameters, these standards define the dimensions and geometry of the specimens to be used during testing.

These test specimens are considered to have a representative elementary volume, meaning it is not necessary to take account of the final geometry of the materials used in the technology. However, the special nature of these tests has required GTT to design some test specimens, adapted to test conditions at cryogenic temperature. The test procedures are adapted to the company's specific requirements.

Off-the-shelf equipment is not sufficient for the measurement of all the characteristics essential to the development of technology for the transport and storage of liquefied gases. GTT therefore regularly develops its own testing facilities, adapted to the requirements of its materials engineers.

GTT's laboratory thus plays a central role and is a key element in the company's process of advancing its knowledge of materials behaviour.

Thermal conductivity equipment

The transport of liquefied gases at cryogenic temperature subjects the technologies involved to a high thermal gradient of approximately 180°C (between room temperature and the LNG temperature). GTT's technologies consist of a succession of layers of materials with different thermomechanical properties. These materials have to be assembled together. The range of materials used and tested is thus very wide. The quantity of equipment available in the laboratory therefore reflects the required characteristics and the diversity of these materials.

Figure 4. Universal testing machine (UTM) image.

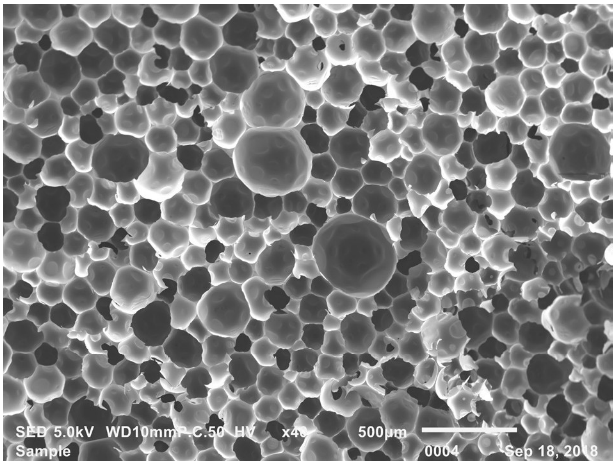


Figure 5. Scanning electron microscope (SEM) image of cells.

The insulation performance of the technology is essential to ensure that the structure of a ship, for example, remains at a permissible temperature for its grades of steel. This thermal insulation performance is also reflected in the daily evaporation rate, or boil-off rate (BOR). GTT offers a range of technologies with BORs between 0.07 and 0.15% (for a standard LNG carrier with four tanks and a 174 000 m^3 load).

The materials input data for BOR calculation is the thermal conductivity (λ). This characteristic is a function of the temperature and is not linear: λ(T).

To measure this value, GTT's non-metallic materials laboratory has different test facilities.

Three guarded hot plates (GHPs) are used to measure the thermal conductivity, by an absolute method, between -160 and 200°C and a pressure range between 1 and 1000 mbar. With this test equipment, it is also possible to reproduce the gaseous environment around the specimens. The dimensions of the specimens are 300 x 300 x 25 mm. A test of this type lasts around 30 hours and requires approximately 1000 litres of liquid nitrogen.

This knowledge makes it possible to choose the optimum insulation material (performance/cost) according to the required BOR.

GTT's non-metallic materials laboratory also has other methods to measure thermal conductivity, such as heat flow meters (HFMs). These test facilities allow for relative measurement between -160 and 40°C at atmospheric pressure.

Another important thermal characteristic to be measured is heat capacity (Cp). This characteristic is also a function of temperature: Cp(T). GTT possesses an adiabatic calorimeter to measure thermal performance between -160°C and room temperature.

An important characteristic of this calorimeter is the ability to carry out this test on large specimens (80 x 10 x 10 mm). Because the specific density of the insulating materials is low, this size is important in order to have a test specimen of sufficient mass to give a precise measurement.

This characteristic also makes it possible to perform thermal calculations and to optimise transient phases, such as the loading and unloading of the cargo.

The coefficient of thermal expansion (α) of materials is also an indispensable characteristic to apprehend. This characteristic is also a function of temperature and is nonlinear: α(T).

With the strong thermal gradient supported by GTT's technologies, the control of the thermal contraction coefficient is important in order to limit thermal stress, and hence increase safety coefficients. The laboratory has various test facilities, including a horizontal dilatometer, in order to measure the α between -160 and 50°C, for example.

Different conditioning chambers allow GTT to store and age materials between -80 and 70°C.

Mechanical and physical equipment

Mechanical stresses are extremely high inside the tank of an LNG carrier, because of its dimensions (40 x 30 x 30 m) and the liquid motion inside. All mechanical characteristics of the materials must also be known. GTT's laboratory has four universal testing machines (UTMs) to carry out mechanical tests on specimens. Coupled with different force sensors, these UTMs allow the characteristics between 0.1 N and 250 kN to be measured. All types of testing can be performed, including compressive, tensile, shearing or bending tests.

These test facilities are complemented by extensometers, specially designed for cryogenic temperature to accurately measure expansion/contraction. GTT's UTMs are equipped with climatic chambers, allowing the company to carry out all of these mechanical tests at between -170 and 23°C.

Liquid nitrogen consumption is approximately 800 litres per day per machine, for testing at -170°C.

Knowledge of mechanical performance as a function of temperature is also essential for the sizing of GTT's technologies. The number of tests therefore has to be multiplied, in order to have perfect knowledge of the materials. The mechanical tests are relatively time-consuming, as it is necessary to ensure that the temperature of the test specimen is uniform and at the desired temperature. To validate this, suitable thermocouples are placed on representative test specimens that are monitored continuously.

The number of specimens tested per day at cryogenic temperature is four times lower than the same tests carried out at room temperature.

The laboratory also has a test bench for Charpy (and Izod) impact testing, with various hammers between 1 and 50 N, and dedicated to polymer materials. A materials database groups these mechanical characteristics as a function of temperature. This data is then used by the calculation department in the development of new technologies.

GTT's laboratory is equipped with numerous physical testing facilities to characterise materials. The latest acquisition is a gas chromatography/mass spectrometry (GC-MS), which, among other things, allows the various gases present in the cells of polyurethane foams to be identified and quantified. These gases are essential for thermal conductivity because they make up approximately 90% of the polyurethane foam. Since each gas has a different λ(T) and thermodynamic laws, optimising this gaseous composition makes it possible to choose the best materials.

Samples are prepared under helium atmosphere, with a glovebox, for easy detection of gases, such as N_2, O_2 and CO_2 also present in air

A scanning electron microscope (SEM) allows high resolution visualisation of the structure of materials with a magnification of x30 to x100 000, to understand the microstructure of materials and the morphological phenomena induced by cryogenic temperature. LNG