characterization

PVT device: characterizing volumetric and linear deformations of composite materials

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At midway between a dilatometer and a calorimeter, the PVT (Pressure Volume Temperature) device provides composite manufacturers with a reliable way to determine the properties of materials to help them model the thermal, chemical and mechanical phenomena that occur throughout their forming processes.

Capacités is a private subsidiary of the University of Nantes that offers research and development services. One of its business units, housed in the Heat Transfer and Energy Laboratory at Nantes (LTEN), acquired a solid expertise in characterization issues related to the thermal properties of composites.

Predicting and controlling dimensional behaviour is indeed the cornerstone of manufacturing high-end thermoset composite parts. Most defects appear during the curing stage, where the material undergoes major deformation due to thermal expansion or contraction and chemical shrinkage.

The coefficients of chemical shrinkage and thermal expansion (CCS and CTE) describe the material deformation induced by both phenomena. Their evolution is driven by pressure and temperature but also depends on the material's state (uncured, cured), which makes their characterization even more complex. Indeed, both these significant parameters and the cure kinetics of the thermoset resin are needed to perform a numerical simulation.

PVT (Pressure – Volume – Temperature) devices were developed in order to address these issues. By monitoring the evolution of the material's volume and temperature during a given curing cycle (temperature and pressure), thermal, chemical and mechanical deformation can be accurately quantified.

Volumetric variation characterization: the PVT-α device

The PVT-α device is a multifunctional system developed by LTEN to perform calorimetric and volumetric analysis simultaneously throughout the curing process of thermoset resins.

Working principle

This device is a steel mould with a 50mm-diameter cavity where a sample (resin or composite) is put under pressure with the help of a piston (Figure 1).

Heating is ensured by several embedded heaters, and cooling by pressurized air circulating within the mould and piston. Heat transfer is characterized by means of non-intrusive heat flux sensors located on each side of the sample. The measurements are analysed to identify the sample surface temperature as well as outgoing heat fluxes, especially during crosslinking.

The variation of the sample volume throughout the curing process is evaluated with an LVDT sensor that measures the piston's displacement with regards to the mould cavity.

The temperature and pressure ranges available are close to the processing conditions of thermoset materials: up to 200°C and 100 bar respectively.

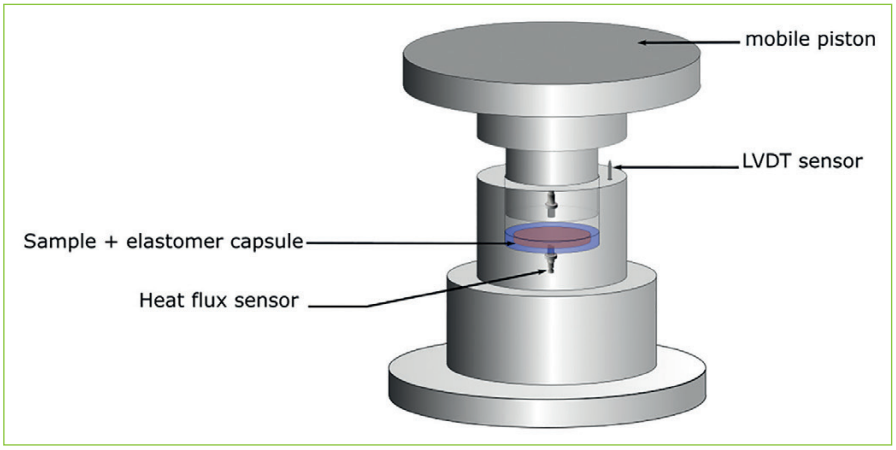


Fig. 1: The PVT-α device

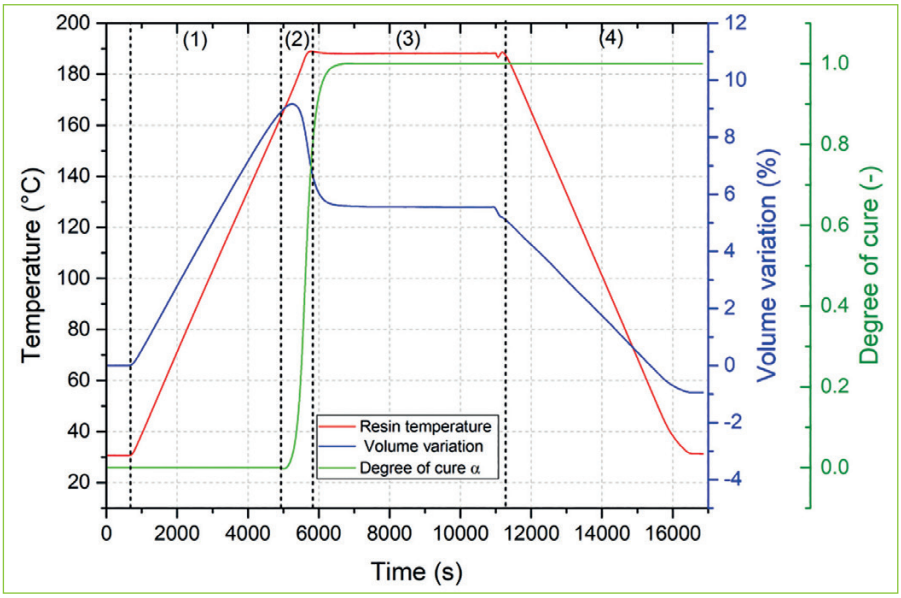


Fig. 2: Relative volume variation of a Hexcel RTM6 resin during processing, along with temperature and degree of cure

Application: Analysing the thermal and chemical properties of RTM6 resin

Figure 2 shows the evolution of the relative volume of an RTM6 resin cured according to the indicated temperature cycle (red curve). The degree of cure, α (green curve), is determined based on the integration of the total measured heat flux as a function of time.

Several steps can be highlighted:

- (1) On the heating ramp, the resin is liquid (α = 0) and the relative volume varies linearly with temperature according to CTEuncured.
- (2) Resin crosslinking is initiated. Consequently, the volume starts to decrease and varies linearly with α according to CCS. Thermal expansion and chemical shrinkage occur simultaneously at this stage, a specific analysis is necessary to separate these phenomena.
- (3) During the isothermal dwell, the volume variation is induced by chemical shrinkage only, as the reaction continues.
- (4) During the cooling ramp, the reaction is complete (α = 1) and the volume contraction is temperature linear with CTEcured.

The sample's volume can be expressed by the following equation:

$$v = (CTE(\alpha)\Delta T) - V_\alpha/V_0 \; CCS \; \Delta\alpha$$

where V_0 and V_α are the resin volume at the initial time and when the reaction begins.

The device's performance was established in many research studies [1]. Various thermoset resins were successfully characterized by Capacités's team for leaders in the aeronautics and automotive industries.

Recently, the features of the PVT-α device were extended to include mechanical characterization. By applying an oscillatory constraint to the sample, the resin's bulk modulus can be identified, as well as its evolution with the degree of cure.

PVT-α is designed to characterize isotropic materials such as resins. However, volume measurement is insufficient to fully describe reinforced thermoset materials, which display an anisotropic behaviour. The experience drawn from the development of the PVT-α helped in designing a new multi-functional device.

Towards the characterization of anisotropic deformation: PVT-HADDOC

The PVT HADDOC (Heterogeneous Anisotropic Deformation and Degree of Cure monitoring) device is designed to characterize the in-plane and out-of-plane dimensional variations of a composite laminate submitted to a cure cycle [2].

The operating principle, shown in Figure 3, consists in positioning a prepreg sample between a steel plate and a mobile piston, placed in a transparent cavity filled by pressurized silicon oil. Hydrostatic pressure can then be applied to the sample.

The through-thickness laminate strain is evaluated from the displacement of the piston, which moves with the sample. Longitudinal deformation is measured with a contactless profilometer scanning the sample's lateral surfaces.

Figure 4 depicts the three-dimensional strains of a Hexcel M21 UD prepreg [3] as well as the degree of cure measured during a four-stage temperature cycle (red curve) similar to the cycle presented in Figure 2.

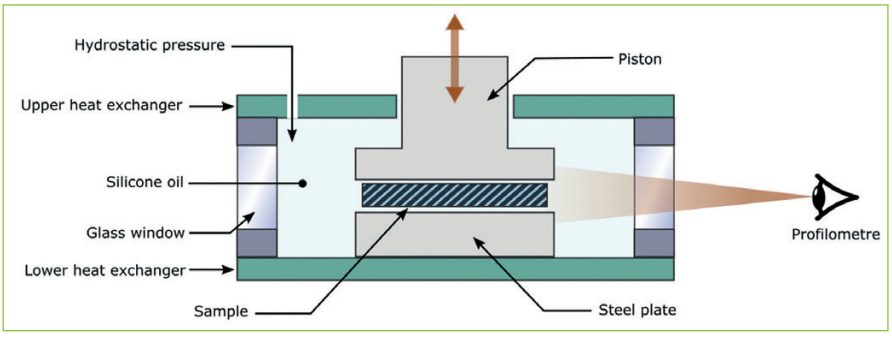
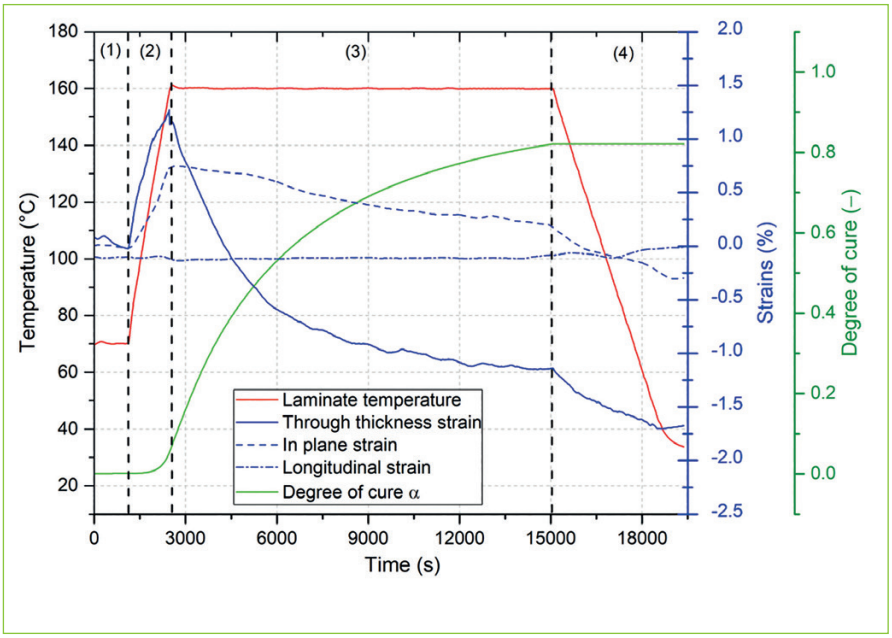


Fig. 3: PVT HADDOC



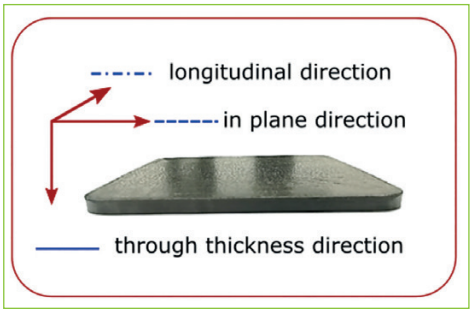


Fig. 4 : Strains along three directions during the curing of an M21 prepreg, along with temperature and the estimated degree of cure

The laminate's behaviour is clearly orthotropic. The longitudinal strain remains mostly unaffected by the thermal and chemical transformations undergone by the composite, contrary to the transverse and through-thickness deformations.

By separating the effect of thermal expansion from chemical shrinkage in each measured strain during stages (2) and (3), the CTE and CCS of the raw composite are successfully estimated in all three directions. Finally, the three-dimensional CTE of the cured laminate is evaluated in stage (4). □

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