



# Investigation into compressive properties of liquid shim for aerospace bolted joints



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## ARTICLE INFO

*Article history:*  
Available online 4 November 2013

*Keywords:*  
Liquid shim  
Compressive testing  
Bulk properties  
Curing process  
Environmental conditioning

## ABSTRACT

Liquid shim is an epoxy-based paste commonly used to fill gaps encountered in composite assemblies, found for example in primary aircraft structures, allowing a better load transfer between mechanically fastened components. In service, liquid shim is exposed to different environmental and mechanical conditions, such as elevated temperature and compression. This paper investigates the compressive properties of liquid shim. Bulk specimens were manufactured, using different manufacturing processes, and an attempt was made to reduce the void content to obtain the optimal mechanical properties of the liquid shim. For example, air entrapment was inherent in the mixing process of the two-part material; this was reduced using centrifugation. The effect of exposure to low ( $-59\text{ }^{\circ}\text{C}$ ) and elevated ( $+85\text{ }^{\circ}\text{C}$ ) temperatures and to  $\text{CO}_2$  was investigated. It was found that liquid shim cured at room temperature did not achieve full cross-linking. In addition, exposure to an elevated temperature of  $+85\text{ }^{\circ}\text{C}$  affected the mechanical properties: there was an increase in compressive strength and a reduction in stiffness. These findings may have important implications regarding the use of liquid shim in certain critical applications.

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## 1. Introduction

Owing to benefits in terms of increased specific strength and stiffness, the use of composite material in aircraft structures is constantly increasing. After being introduced progressively in secondary structures, composites are currently adopted in primary structures (e.g. fuselage, wings, and empennage) of recent programmes, such as the Boeing 787 or the Airbus 350 XWB [1]. In the latter, this leads to a proportion of composite material of 53% by weight. The rest is made up of aluminium alloys (19%), titanium (14%) and other materials (14%) [2]. Primary structures such as wings have a carbon fibre skin, while the ribs are usually made from aluminium alloy. Many solutions exist to join composite panels to the metallic structure. Bonded joints simplify the manufacturing process and are lightweight, but in most cases a mechanically fastened solution is chosen, owing to their reliability and to allow for the possibility of disassembly for inspection or replacement of damaged components [3]. Due to surface irregularity of the composite structure and the problem of assembly tolerances after the component manufacture, gaps can occur at the faying surfaces. Because of the brittle behaviour of the composite, the elimination of such gaps cannot be resolved during the assembly process by the clamping force of the fasteners alone: this would result in the development of cracks or delamination in the composite

material and would compromise the performance of the structure. To prevent damage, gaps have to be filled. This results in better mating surfaces, producing more optimal load transfer in the joint. It is recommended to use liquid shim to fill gaps between 0.13 mm and 0.76 mm, and to use a combination of solid and liquid shim for gaps beyond 0.76 mm [4–6]. Liquid shim, which is a thixotropic epoxy-based paste, was developed in response to this need. This material is ideal for application over large surfaces and can be cured at room temperature. Once cured, it has high compressive strength and resistance to micro cracking [7].

For the fabrication of the joints, liquid shim is usually applied to one part and covered with a release film for the curing process, so that the shim is bonded to only one substrate, allowing disassembly. The joint is then mechanically fastened. In service, such joints can be exposed to severe mechanical and thermal conditions. Hühne et al. [8] studied the structural behaviour of shimmed joints. The performance of liquid shim in themomechanical fatigue was analysed by the authors in a previous study [9]: single-lap joints were exposed to mechanical fatigue loading at low ( $-59\text{ }^{\circ}\text{C}$ ) and elevated temperatures ( $+85\text{ }^{\circ}\text{C}$ ), representative of aircraft service conditions ( $\text{CO}_2$  gas was used as the cooling medium). An evolution of the stiffness of shimmed joints was observed at the first exposure to elevated temperature. It was also shown that the liquid shim was subjected to significant compressive loads due to the bolts' clamp-up force and secondary bending, which occurs in single-lap joints [10]. Following on the abovementioned study, characterisation of the compressive properties of the liquid shim

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material and the effect of environmental conditions was undertaken. This work may provide input to the design and numerical analysis of such joints.

If the high viscosity of the liquid shim is an advantage for its industrial use, allowing it to be dispensed on inclined surfaces, it is more problematic as regards air entrapment even if precautions are taken to minimise it. Dispensing cartridges are designed to avoid the creation of air pockets while filling [11], but voids still could be contained in the adhesive when supplied. Mixing of two-part adhesive systems or casting can also introduce air into the adhesive. The use of a static mixer seems more efficient in terms of reduction of air entrapment during mixing, but some adhesives are also supplied in Semkit® form where the two parts are separated with a barrier and mixed with a plunger. Several methods have been tested to remove air from adhesives, such as degassing under vacuum prior to casting, centrifuging [12], mixing by hand under vacuum or heating the resin to reduce the viscosity when possible [13]. However, these techniques may be inappropriate or too expensive to be set up in an industrial environment where production time and costs have to be minimised. Also, there is a pronounced difference between the fabrication of thin layers of adhesive, i.e. around 1 mm thick, and the preparation of bulk specimens such as standard rods for compressive testing [14]. In the latter, the presence of small voids would not significantly affect compressive properties of the bulk specimen, but samples have to be free of large voids, which could lead to stress concentration and premature failure. To overcome this, centrifuging methods have been successfully used to produce bulk samples [15]. Void characterisation is commonly performed using X-ray scanning techniques [16] or analysis of micrographs using thresholding techniques to differentiate the voids from the material [17].

Finally, environmental conditions to which liquid shim could be exposed, e.g. elevated/sub zero temperatures or gas [9], could have an effect on the properties of polymeric materials. It is known that the sorption of carbon dioxide in polymer networks can lead to a plasticisation and changes in mechanical properties [18,19]. Some studies of two-part epoxy based materials revealed that the curing process at room temperature was not complete, even after several days [20]. In this case, dynamic mechanical analysis (DMA) was successfully used to study the state of cure of epoxy-based systems [21,22].

The primary objectives of this paper are to characterise the mechanical behaviour of liquid shim material in compression and to study the effect of environmental parameters such as temperature and exposure to CO<sub>2</sub> on the residual compressive properties. Different methods were investigated concerning the manufacturing and curing processes of bulk liquid shim specimens. A secondary objective was to investigate air entrapment in the material during the manufacturing process.

## 2. Materials and methods

### 2.1. Material

The shim material used in this study was the third generation liquid shim Hysol EA 9377, manufactured by Henkel. It is a thixotropic epoxy based adhesive supplied in a Semkit® dispenser, which contains two separated components to be mixed: part A, which composed of thermosetting resins (epoxy and epichlorohydrin-4,4'-isopropylidene diphenol), silica components, and carbon fibre [23]; and part B, which composed of curing agents [24]. The pot life is 60 min after mixing and the recommended normal curing time is 5–7 days at 25 °C.

### 2.2. Manufacturing of test specimens

The cylindrical bulk liquid shim specimens were manufactured using an injection moulding technique. The material, stored at 4 °C in a refrigerator, was removed at least 1.5 h before use, allowing it to reach room temperature before injection. The two parts were manually mixed in the cartridge using the plunger rod in accordance with the manufacturer's instructions, i.e. 100 strokes within 4 min. A special nozzle (Fig. 1) was screwed into the neck of the cartridge. The nozzle allowed a gentle transition for the liquid shim to flow between the cartridge and the mould. Due to the exothermic curing reaction of the adhesive, the moulds were made of steel tubes (inner diameter of 12.7 mm) to mitigate any significant rise in temperature. The mould inner surface was firstly cleaned with acetone. Secondly, to facilitate the release of the specimens, a release agent (Airtech Safelease 30, a water based PTFE liquid) was applied twice with lint-free swabs, and allowed to dry before injection of the shim material. The cartridge was mounted in a pneumatic gun (supplied by Loctite) to dispense the liquid shim. Moulds were placed at the end of the nozzle and the adhesive was then injected into the moulds (Fig. 1). Once full, the moulds were detached from the nozzle and placed on a release film.

As some voids were assumed to be present, an X-ray system (Hewlett–Packard Faxitron series, used at 80 kV and 2.5 mA, combined with an EZ 240 scanner, from NTB elektronische Geräte, Germany) was used at different stages of the manufacturing process to evaluate the quality of the material in terms of void content. This equipment was considered suitable as the detection of voids bigger than 1 mm were targeted. The detection of such voids was validated by the scan of a reference sample, i.e. a bulk liquid shim cylinder containing a defect of known dimensions (1 mm diameter hole).

Several cases (batches) were considered to evaluate the effect of the curing process and conditioning parameters on the mechanical properties of the material:

- Batch A: Cured at room temperature for a minimum of 5 days (typical production process).
- Batch B: Cured at 80 °C for 90 min in a hot drape former (accelerated cure cycle).
- Batch C: Cured at room temperature under pressure (void minimisation strategy). A constant load was applied to the material through a piston placed at the top of the mould. A porous peel ply was placed at the bottom of the specimen to allow air to escape. A pressure of 0.8 MPa was applied.

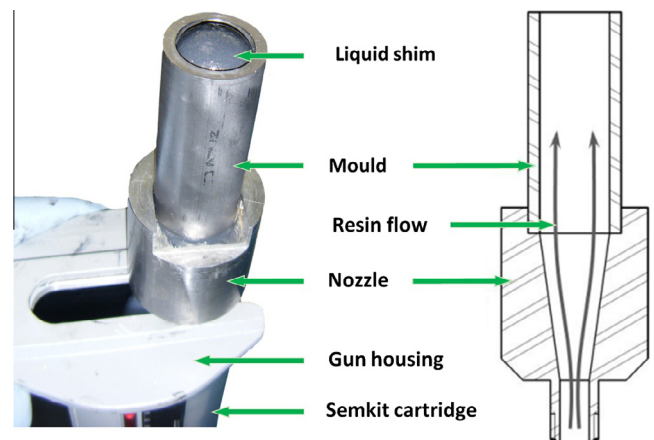


Fig. 1. Illustration of the injection process of liquid shim into the mould.

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