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Pressure-assisted infiltration of molten metals into non-rigid, porous carbon fibre structures

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ABSTRACT

Mercury intrusion porosimetry has been conducted on a range of non-rigid, porous carbon fibre structures. Comparison with data from gas pressure infiltration experiments in a molten Al-Si alloy shows it to be a useful tool in determining the pressure required to produce Al metal matrix composites with low levels of porosity. Whilst for non-rigid fibre preforms, as studied here, it is difficult to pin-point every aspect of the infiltration process, the method does give an indication of critical aspects of the infiltration behaviour. Preforms made from loose and spread tow can be fully infiltrated at relatively low pressures (12 bar) and although metal can fill the spaces between fibre bundles within textile-based preforms at low pressures, densely-packed bundles are only infiltrated at higher pressures, usually in excess of 50 bar. Mercury intrusion porosimetry could provide a valuable and simple tool in the design of fibre-reinforced metal matrix composites with optimised structures that are easy to manufacture.

1. Introduction

Infiltration processing is a versatile and cost effective method for the production of metal matrix composites (MMCs). By this procedure, molten metal is forced, using either mechanical or gas pressure, into the interstices or channels within a porous structure, commonly comprised of rigid packed beds of particles or fibres, with the aim being to fill all the available pore space and produce a dense product.

For polymer composites, where wetting between the resin matrix and fibre reinforcement is generally good, the key role of the applied pressure is to facilitate rapid transfer of high viscosity resin throughout the network of channels between and within the fibre bundles in the preform. Although Kennedy et al. (2000) showed, by example of spontaneous wetting of ceramic powders by molten Cu, that wetting can be good in some metal-ceramic systems and Gil and Kennedy (2012), that spontaneous wetting is also possible in Al-systems under certain tailored atmospheric conditions; generally, under normal processing conditions, wetting between Al alloys and most candidate ceramic reinforcements is poor. Kennedy and Karantzalis (1999) presented a large number of examples to confirm this, relating the non-wetting behaviour to contact angles that greatly exceed 90°. Despite low melt viscosities for molten metals, poor wetting necessitates the requirement for an external pressure to be applied for infiltration to take place. Being able to predict the pressure required to initiate the intrusion of liquid metal into a porous preform and the maximum pressure required to eliminate all the porosity, is paramount to designing preform structures and the apparatus required to affect infiltration.

Mercury intrusion porosimetry (MIP) can be used to generate plots of the cumulative volume of liquid intruded into a rigid porous structure as a function of the pressure applied. These plots are characteristic of the pore size and geometry of the porous structure as well as intrinsic wetting characteristics for the system. These data can be readily converted into drainage curves; plots of the degree of filling (or saturation) of the porous structure, against pressure. Bahraini et al. (2005) used a bespoke high temperature porosimeter to demonstrate that drainage curves for a given porous structure, derived from conventional Hg porosimetry (at room temperature) are of the same form as intrusion by molten metal at high temperature, since the infiltration paths are self-similar from one metal to the other, the only difference being the intrinsic wetting behaviour. Conversion from the Hg (MIP) to a molten Al system is then achieved by multiplying the Hg data by a scaling factor, φ, the ratio of the wetting behaviour (the magnitude of the work of immersion) in the Al-preform and Hg-preform systems. This factor is described by:

$$\phi = \frac{\cos(\theta_{Al-Pre})\sigma_{LV^{Al}}}{\cos(\theta_{Hg-Pre})\sigma_{LV^{Hg}}} \quad (1)$$

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where σ and θ are the surface tension and contact angle for the respective systems. This relationship has been shown to hold for rigid packed beds of particles of different morphologies, for materials including alumina and diamond (Bahraini et al., 2005), AlN (Kida et al., 2008), graphite (Molina et al., 2007) and SiC (Bahraini et al., 2008).

Carbon fibres (CF) are ideal reinforcements for Al matrices as their high stiffness and low density offer the potential for achieving significant increases in the specific stiffness for the composite, over the base material. Carbon fibre composite fabrics are derived, in the main, from mm-sized bundles of carbon fibres (tow) which contain thousands of tightly-packed 5–10 µm diameter individual fibres. Research and development in carbon fibre polymer composites has seen the creation and implementation of diverse 2D and 3D fibre architectures to produce tailored mechanical properties, but the design of MMCs with CF reinforcement draws very little from the experiences in polymer systems, using mainly simple uni-directional and 3D random architectures.

This study applies MIP to a range of diverse CF architectures developed for wettable polymer resin systems, with the distinction from previous studies that these materials are not rigid structures nor do they possess simple pore geometries (unlike, for example, as achieved in packed powder beds). MIP observations are compared with data from Al-CF infiltration experiments at a range of pressures, with the aim being to establish whether this approach can be used to accurately predict the infiltration behaviour for molten Al within non-rigid structures containing multi-scale and complex pore structures and highlight any potential issues for using such products to produce Al-MMCs.

2. Experimental procedure

A selection of different CF preform architectures was used. These are listed in Table 1 and shown in Figs. 1–4. A recycled carbon fibre (rCF) mat is shown in Fig. 1, it is a non-woven, thin mat, made from offcuts of SGL's 50 K (a tow containing 50,000 fibres) non-crimp fabric. The chopped sections of fabric are "fluffed up" separating the fibre bundles, and then needle-punched to produce a widely-spaced mixture of randomly-orientated individual and loose discontinuous bundles of fibres, typically 60 mm in length with an approximate volume fraction of 0.6.

Fig. 2a shows a preform fabricated by crochet (Cro-CF). Crocheting is a process that uses a hook to fabricate a fabric from a continuous length of CF tow by creating loops. The crochet hook was 5 mm in diameter, which determined the size of the loops. Toray T700-60E 12 K carbon fibre tow was crocheted into a flat strip with an approximate overall fibre fraction of 0.29, as is shown in Fig. 2b.

A carbon fibre plain weave (WCF) was investigated, consisting of 6 K carbon fibre bundles in the warp and weft directions, as shown schematically in Fig. 3a, producing a mat with a fibre fraction of approximately 0.60. A 3D woven CF (3DWCF) preform was also studied, shown in Fig. 4. The through-the-thickness weave had a binder-to-warp-stack ratio of 1:1, i.e. each binder tow was separated by one vertical stack of warp tows. This material contained an alternating stack of 9 weft layers and 8 warp layers, forming a dense 3D fabric that was approximately 3.5 mm thick. The carbon fibre tows were AKSACA A-38 with 6 K filaments for the warp and weft tows, and 3 K filaments for binder tows. The fabric had an overall fibre volume fraction of approximately 0.55 and repeating units that are on a finer scale to that for the WCF material.

Table 1
CF preform types used to fabricate MMCs.

Preform	Fibre type	Construction	Tow size
rCF	Recycled (60 mm fibre length)	Non-woven	N/A
Cro-CF	T700SC	Crocheted	12 K
WCF	HTS40	Plain weave	6 K
3DWCF	ASKACA A-38	3D weave	6 K warp/weft 3 K binder

CF prefoms were infiltrated using a gas pressure infiltration method (GPI), at a series of different pressures. Preforms were fabricated by die-cutting circular discs of diameter 28 mm from each of the CF fabrics, and stacking multiple discs within a 29 mm diameter alumina crucible (an example of stacked rCF fabric discs was shown in Fig. 1c). The Cro-CF mas was rolled into a cylinder, as was shown in Fig. 2c, before being loaded into the crucible. The Cro-CF, WCF and 3DWCF preforms weighed approximately 10 g, but owing to the poor packing between the layers (loftiness) in the rCF preform, only 1.7 g could be loaded into the crucible.

Approximately 60 g of an Al–12 wt.% Si alloy was placed on top of the preforms and the assemblies were placed inside a bespoke pressure vessel. The vessel and its contents were heated to 550 °C under vacuum for 1 h to remove sizing from the preforms, and then raised to 750 °C and held for 1 h to allow the Al to melt and to form a liquid metal "seal" over the preform. Thereafter, Ar gas pressure was applied (at pressures ranging from 0.5 to 55 bar ± 5%) for 30 min, followed by cooling and solidification under pressure. The GPI rig and a schematic of the internal setup are shown in Fig. 5. MMCs were sectioned and mounted in conductive resin, polished to a 1 µm diamond finish and studied using an optical microscope.

MIP data were obtained using a Micrometrics Autopore 4 Hg Porosimeter, at mercury pressures from 0.06 to 100 bar, using the largest available penetrometer size (25 mm in diameter and length). Samples were prepared in the same way for the penetrometer as for infiltration with Al, but using a 22 mm diameter cutter.

3. Results and discussion

Data before the onset and at the end of MIP measurements yield values for the bulk and apparent densities of the preforms. The bulk density indicates the amount of uninfiltrated space within the preform, influenced by the separation between the layers in the multi-layered structure and any compression of this structure caused by the pre-stress from the Hg in the penetrometer (approximately 0.006 MPa). The apparent density, taken at the end of the pressurisation cycle, should, if all the porosity is interconnected and the preform is fully infiltrated, be equal to the density of the carbon fibres.

Despite the relatively high fraction of fibres in the rCF mat, the preform fibre fraction, calculated from the bulk density, was only 0.12, demonstrating the high degree of separation between the layers (loft) prior to infiltration. The fibre fraction estimated from the bulk density in the Cro-CF sample was 0.52, indicating either some compression of the preform when first filling with Hg or, more likely, permeation of Hg into the void spaces within the preform, via the large pores (loops) that are unique to this structure. The bulk densities for the WCF and 3DWCF samples were 0.50 and 0.62 respectively (compared with 0.60 for the WCF mat and 0.55 for the 3DWCF), suggesting some separation between the layers in the multi-layered WCF preform and a small degree of compression of the 3DWCF preform at the minimum Hg pressure. In all cases, the apparent density for the preforms, taken at the maximum intrusion pressure (100 bar) was greater than 99% of the estimated fibre density, indicating, within experimental error, that complete infiltration (saturation) had taken place.

Fig. 6 shows the MIP data for intrusion with Hg, normalised with respect to the maximum volume intruded, giving the fraction of saturation as a function of pressure. Each of the plots exhibits a linear region at lower pressures, which is a result of compression of the preforms before infiltration takes place. These regions are more evident for the more compliant structures (those with steeper gradients of saturation vs. pressure) such as the rCF and WCF preforms, which show similar behaviour, compressing readily as the spaces between the thin, lofty, multi-layered laminates are removed. The 3DWCF sample, made