Powder Technology 246 (2013) 284–302

Contents lists available at SciVerse ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Characterisation and replication of metallic micro-fluidic devices using three different powders processed by hot embossing

M. Sahli *, J.-C. Gelin, T. Barriere

Femto-ST Institute, Applied Mechanics Dept., CNRS UMR 6174, ENSMM, 25030 Besançon cedex, France

ARTICLE INFO

Article history:
Received 31 July 2012
Received in revised form 3 March 2013
Accepted 14 May 2013
Available online 23 May 2013

Keywords:
Hot embossing
Thermoplastic binder
Micro-structures
Polymers
Feedstocks

ABSTRACT

This study presents the results of micro-replication of different powder feedstocks samples. The feedstocks of metal powder and plastic binder with various solid loading in the range from 40% to 75% were prepared. The different feedstock comprises 316 L stainless steel, Fe – Ni 8%, or WC-Co and a multi-component binder system. Suitable polymers/powder formulations for the hot embossing were chosen and characterised by thermogravimetric analyses (TGA), mixer measurements and capillary rheology testing in order to check and quantify the viscosity and homogeneity of all the developed feedstocks. The photoresist (SU-8) was used as a structural material that was deposited onto the Si wafer to obtain the micro-fluidic systems with low roughness. Then the elastomeric mould was obtained using casting with one face that contains all the details of the original master, very well defined and smooth sidewalls.

The objective of the paper is to develop metallic micro-structured die mould cavities with higher quality using hot embossing process with various different powder loaded polymers. However, a rapid prototyping process was adapted for fast and cost-efficient manufacturing. This study compares the capacities of three different metallic powders to emboss of the metallic replications by hot embossing, the dimensional stability, the mechanical strength and particularly hardness of sintered components. The results show that the feedstock can be used for the manufacturing of the micro-fluidic die mould cavities with a low roughness, proper dimensions and good shape retention.

1. Introduction

In recent years, the miniaturisation and manufacturing of micro-components have gained increasing attention in both industry and academia [1–4]. The microinjection moulding process is rapidly becoming one of the most promising fabrication technologies for the mass production of thermoplastic polymer micro-parts [5–7]. Most of the applications are concentrated in the fields of micro-optics, microfluidics and medical instruments. Hot embossing is also a manufacturing method used for the fabrication of polymer-based micro-components. It provides several advantages, such as low-cost moulds, high replication accuracy for micro-feature generation and ease of operation [8,9]. For these reasons, manufacturing by hot-embossing [10,11] rather than injection moulding is advantageous. It also introduces less residual stress into the polymer because the polymer chains must stretch and flow only a very short distance from the substrate into the patterned microstructure during hot-embossing. Additionally, the temperature variation range for the polymer is smaller than that required in injection moulding, which leads to reductions in both the shrinkage that occurs during cooling and the frictional forces that act on the micro-features during de-moulding [12–16].

Conventionally, the mould tooling is made of either steel or aluminium and can be manufactured by Computer Numerical Control machining (CNC) and Electric discharge machining (EDM); after it is manufactured, the mould can be used to produce polymer micro-parts via either hot embossing or injection moulding [17,18]. The surface quality of the mould is highly dependent on the cutting conditions used during machining, which significantly influence the resulting surface roughness [19,20]. For example, in micro-fluidic systems, these techniques generate tools with a high surface roughness and micro-channels that generate a reduction in the overall efficiency of fluidic flow. In this study, to establish the basis of micro-pattern manufacturing of the metallic tool using the hot embossing process, an alternative method was applied to form micro-channels in the metallic moulds with a low surface roughness. The micro-fluidic chip developed here is used as an example to discuss the use of various mould insert materials by hot embossing.

Various investigations have been conducted concerning the influence of both the physical and processing parameters, such as the particle size and solid loading of the powder in the mixture, the effect of sintering temperature on the control and dimensional changes, the density, mechanical properties and roughness surfaces of the components, have received increasing attention in recent years [21]. German et al. [22] investigated the effect of solid loading on the dimensional change that occurs during solvent debinding of the injection moulded

* Corresponding author. Tel.: +33 381666040.
E-mail address: Mohamed.Sahli@ens2m.fr (M. Sahli).

0032-5910/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.powtec.2013.05.026

components. Liu et al. [23] investigated the effects of solid loading and extrusion on mixing and feedstock homogeneity for the injection moulding of micro-structured components. The results demonstrated that 316 L stainless steel feedstock with a suitable solid loading (equal to 58 vol.%) can be successfully used for the forming of micro-structured parts with proper shape retention. The micro-structured parts exhibit the necessary strength for demoulding with no observable debinding defects and a final sintered microstructure characterised by shape retention.

Recently, Tay et al. [24] have succeeded in the manufacturing of micro-gears using the micro-powder injection moulding process with 316 L stainless steel powder possessing particle sizes of 2.4 μm and a multi-component wax-based binder system. The analysis revealed different grain structures at the tooth (≈35 μm) and hub of the micro-gear (≈5 μm). Significant grain growth was also observed at the tooth. Meng et al. [25] conducted an experimental analysis on the replication of a micro-fluidic system by micro-powder injection moulding using 316 L stainless steel. Additionally, they also investigated the dimensional change and surface roughness of the micro-mixer. They obtained proper replication with appropriate shape retention lacking in visible defects by use of a powder injection moulding process with a 316 L stainless steel feedstock. The dimensional shrinkage of the micro-mixer occurred mainly in the sintering step, whilst the dimensional change was not noticeable in the debinding step. The surface topography of the silicon mould insert was properly replicated in the stainless steel micro-mixer. Fu et al. [26] investigated the manufacturing of a 316 L stainless steel cylindrical micro-structure array using a silicon mould insert with the hot embossing process. Their results demonstrated the effects of various embossing parameters on the filling of micro-cavities in the silicon mould insert and the de-moulding of micro-structures. This study was only focused on the replication of a micro-structured array with an aspect ratio of 2. It was limited to the use of 316 L stainless steel with unknown solid loading and binder percentages in its feedstock. Most of the work up to date on hot embossing focused on to use this process as a flexible, low-cost micro-fabrication method for polymer microstructures over large surface areas. To date our work was focused on the use of the hot embossing process adapted to metal powders for the manufacturing of a micro-fluidic die mould with low surface roughness. An integrated rapid prototyping chain was established for rapid manufacturing of the micro-structured master in metallic powders made from elastomeric moulds by use of the hot embossing process. This rapid manufacturing chain, offers a rapid and flexible manufacturing route for fabrication of prototype micro devices from polymer solutions. This study uses the lithography technology to fabricate the silicon mould insert with nanoscale details and surface roughness at a lower cost. Due to the mechanical strength limitation, silicon material is usually not suitable to use as mould material. In this study, the silicon master was used for casting of silicone to fabricate elastomeric micro-fluidic mould. The choice to use a flexible mould was related to the de-moulding facilities for the micro-structures and the increase of lifetime of the mould when used in the hot embossing process.

This paper describes the elaboration and characterisation of different feedstocks for use in metallic hot embossing and the manufacture of micro-structures with low surface roughness. In the experimental tests presented here, only the temperature and pressure were considered while analysing the manner in which the metallic die master can be archived with the highest replication accuracy. These experiments allow us to understand and qualify the influence of hot embossing processing conditions on the geometrical embossed accuracy of the replicas and the mechanical properties of the final components after solid state sintering. The sintering stage was carried out in vacuum using the metallic micro-structured samples obtained at different solid loadings. Therefore, the present work also analysed the shrinkages, densities, Vickers hardness values and roughness variations of the micro-fluidic specimens using three shades.

2. Experimental materials and methods

2.1. Materials

Fine powders of 316 L stainless steel were used to develop the mixtures dedicated to the hot embossing process. The powder particulates had a spherical shape and an average particle size of 5 μm–80%. This shape is generally more appropriate for obtaining a feedstock with low viscosity. The powders had a density equal to 7.9 g.cm^{-3} and were provided by Sandvik Osprey Company. Fig. 1 gives a photograph of the 316 L stainless steel powder particle distribution.

The micron-size pre-alloyed powder (Fe – Ni 8%) was composed of PA-FN08 and cobalt tungsten carbide with 19.9% WC (Co-WC). The ME1107 powders were characterised to have an average particle size of 5 μm. The powders were provided by Eurotungstene Company® (Eramet) with densities equal to 7.93 and 9.74 g.cm^{-3}, respectively. The powder morphology is given in Fig. 2. Before mixing, the powders were dried in a vacuum oven at a temperature of 120 °C for 5 h to remove any moisture.

Feedstocks of Co-WC, Fe – Ni 8% and 316 L stainless steel based on 40 to 75% solid loadings (by volume) were prepared using a twin-screw mixer. The binder system used in this study consisted of paraffin wax, polypropylene and stearic acid. The composition of the binder corresponded to the ratio of PP:PW:SA given as relative fractions 40:55:5. The characteristics of the different binder systems and the raw powders are related in Tables 1 and 2.

2.2. Processing steps

The main steps of the hot embossing process include the Lithographie, Galvanoformung, Abformung (UV-LIGA) process for the SU-8/Si mould

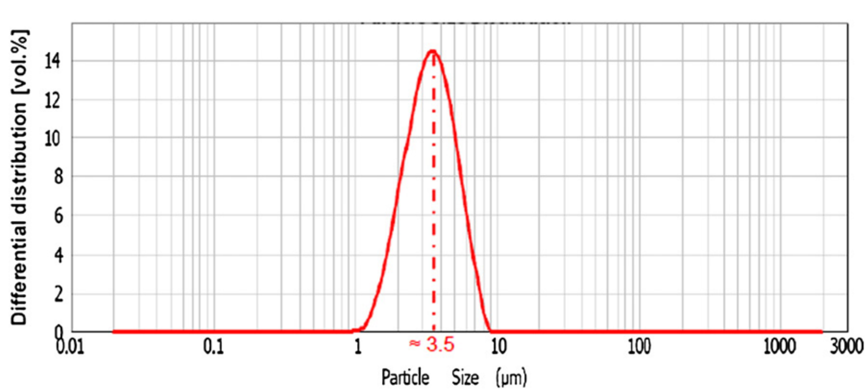

Fig. 1. The particle size distribution for the 316 L stainless steel powder (d_{50} = 3.4 μm).