

RAPID PROTOTYPING OF 3D MICROSTRUCTURES BY DIRECT PRINTING OF LIQUID METAL AT TEMPERATURES UP TO 500°C USING THE STARJET TECHNOLOGY

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ABSTRACT

We present a novel approach for 3D-prototyping of porous metal structures by direct non-contact liquid metal printing, based on the StarJet technology [1]. In contrast to our previous work, the presented droplet generator features an improved nozzle chip design and actuator housing that allows operation at temperatures up to $T_{\max} = 500^{\circ}\text{C}$ (formerly $T_{\max} = 250^{\circ}\text{C}$). This enables the ejection of single droplets of metals with higher melting points like for example magnesium or zinc alloys like ZAMAK. The droplet generation frequency could be increased by a factor of 10 to $f_{\max} = 4$ kHz. Furthermore, deviations of the droplet trajectory from the symmetry axis of the nozzle are reduced to $\Delta\text{deg} = 0.28^{\circ}$ by the new design. This paper reports on experimental results obtained with the improved device and presents 3D metal structures with various porosities.

KEYWORDS

Liquid metal, micro droplets, contactless printing, high melting, StarJet, solder printing, rapid prototyping, porous metal structures

1. INTRODUCTION

The generation of micro droplets of liquid metals is a challenging area in the field of MEMS technologies. It can be used for the generation of solder bumps [3] for flip chip bonding or for rapid prototyping of electric circuits [4] and metal structures [6]. However, the dispensing of hot metal micro droplets is challenging for several reasons: First of all the dispenser must operate at temperatures above the melting point of the metal. Therefore, all parts of the device must be either fabricated of temperature stable materials or have to be thermally insulated from the hot parts of the system. Especially piezo actuators must be isolated [3] or actively cooled to prevent their destruction. The operation temperature of these materials is limited by the Curie temperature, typically ranging between 150°C and 300°C [5]. Also the mechanical stress induced by heating up or cooling down the dispensing device, requires certain design rules to be considered e.g. material combinations with suitable modulus of elasticity and appropriate coefficients of thermal expansion. Furthermore, the molten metal inside the device as well as the ejected droplets have to be prevented from oxidation by the application of protective gas. The StarJet technology [1] used for the dispensing of liquid metals droplets in the presented work is based on a pneumatic actuation principle. The gas flow which works as actuation mechanism avoids the oxidation of the liquid metal in the reservoir inside the device as well as the

dispensed metal droplets in flight. The entire device consists of a pneumatic actuator module made from brass and a star shaped micro nozzle fabricated by silicon micromachining. All connections are sealed by high temperature stable materials e.g. Nova Mica. Thus, the limitation for the maximum operating temperature is the melting point of the actuator material itself only. In consequence the StarJet technology should be suitable for generating liquid metal droplets of all kinds of metals with high melting points without of suffering from the aforementioned problems. Such droplets can be used to produce metal structures by rapid prototyping based on direct metal printing. In particular the digital printing mode allows for the creation of structures featuring various porosities. One application for such porous metal structures is e.g. the fabrication of master structures for the thermo-/cold-forming of thin and flexible, micro-structured polymer films for the production of lab-on-a-foil devices [2]. The porosity of the metal master structures is mandatory in this case to enable the degassing during the moulding process. This work is aiming towards the 3D-prototyping of such porous micro structured masters by demonstrating the direct printing of single metal micro droplets at temperatures up to 500°C .

2. CHIP FABRICATION

The working principle of the StarJet droplet generation method is based on the star-shaped nozzle chip (see fig.1) described in detail elsewhere [1].

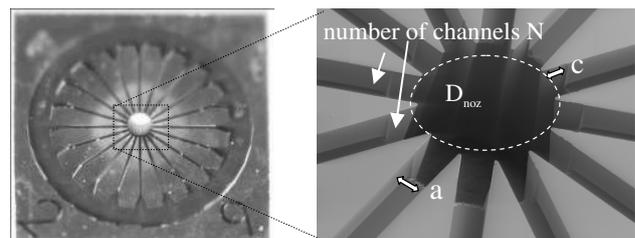


Figure 1: a) Photo of StarJet nozzle chip b) cross section of the star shaped nozzle.

In this work the fabrication process of the chips by deep reactive ion etching (DRIE) has been improved to carve the star-shape structure into a silicon wafer (thickness $t = 300 \mu\text{m}$). The developed two-side etching process P2 (see fig.2) enables the control of the etching depth and prevents from over-etching and charging effects. The individual process steps are shown in figure 2. 1. The front of the Wafer was covered with a $5 \mu\text{m}$ thick resist layer (AZ 4533) that was structured with the geometry for the gas bypass channels 2. The channels were etched ($t = 100 \mu\text{m}$) into the wafer by DRIE etching.

3. The wafer was cleaned and the lower side of the wafer was covered with a 10 μm thick resist layer (AZ 9260) which was structured with the profile of the nozzle channels. 4. The nozzle structures were etched through the whole wafer by DRIE etching. Finally, the resist was stripped and individual chips were separated by dicing.

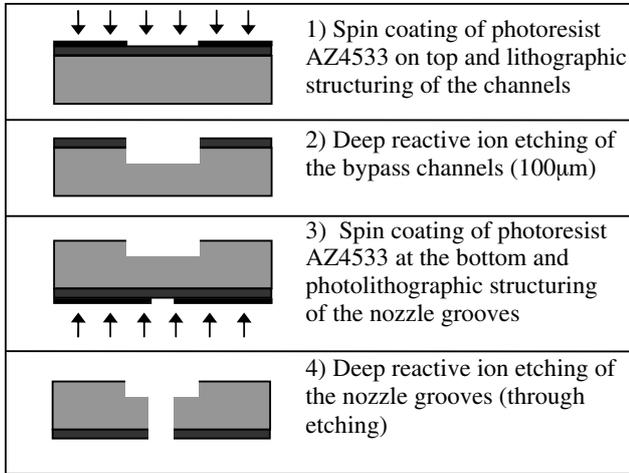


Figure 2: Fabrication process of a StarJet nozzle chip

With this process an improved smoothness and straightness of the structures could be realised compared to the process reported in [1] (see fig.3).

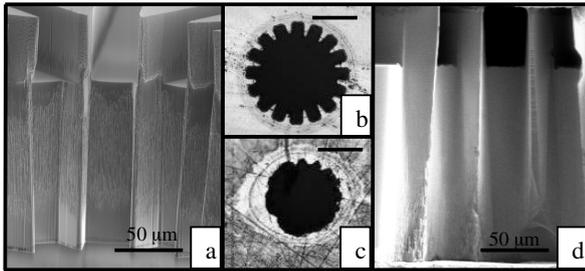


Figure 3: Cross section (a) and bottom view (b) of the improved star shaped nozzle chip. In comparison to the bottom view (c) and cross section (d) of the process from [1]; scale: 100 μm

The used mask consist of 34 individual chip designs defined by the parameters a , c , d_{in} and N as shown in fig. 1. The geometrical parameters of the chips used in this work can be found in table.1.

Table 1: Parameters of StarJet nozzles studied.

	#06	#12	#22
N	12	12	16
a [μm]	20	40	20
c [μm]	40	60	60
d [μm]	89	183	144

4. EXPERIMENTAL SETUP

The improved StarJet actuator (see fig.4) is made of brass and has a maximum outer diameter of 22 mm and a total height of 70 mm. The nozzle chips are connected to the heatable reservoir outlet by a supply channel and are aligned by a laser fabricated positioning spring and

sealings. An external connection (c.f. fig.4 “pressure supply”) at the top of the reservoir allows for the connection of a Nitrogen source to drive the droplet ejection and to rinse the nozzle channels with protective gas.

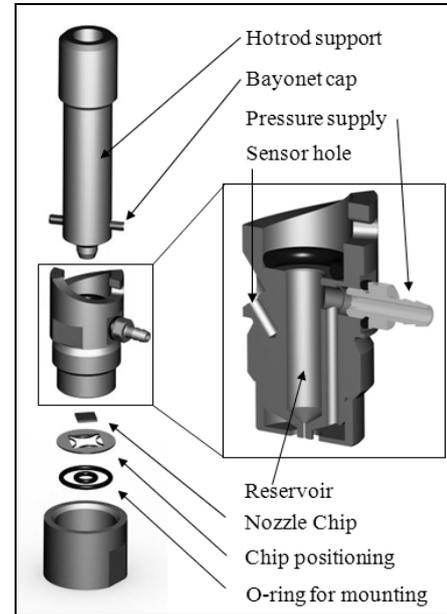


Figure 4: StarJet actuator prototype

An external solenoid valve regulates the gas flow of the nitrogen for pneumatic actuation. It switches between a low nitrogen pressure (20 hPa to 50 hPa) to rinse the nozzle and a higher actuation pressure (150 hPa to 1000 hPa) initiating the droplet ejection. The reservoir is covered by the heater support which holds a 100W heating element (HotRod , Hotset). The heater can be removed by a bayonet cap for an easy refill of the reservoir. The reservoir temperature is monitored by a NiCr-Ni thermopile sensor mounted into a bore close to the reservoir. The measured temperature is used for a closed loop controlled temperature regulation. The temperature control as well as the valve control is realised by a self - developed electronic control unit which is connected to a PC via USB port. This unit allows for the precise, time defined actuation of the valve in combination with the autonomous control of the reservoir temperature.

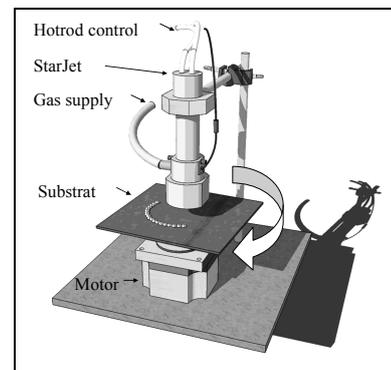


Figure 5: Sketch of experimental setup for tube printing

The setup for the experiments presented in this paper consisted of the described StarJet actuator mounted above a rotating substrate (see Fig 5). The distance between the nozzle and the substrate was constant at $h = 20$ mm and could not be regulated during an experiment. Thus, the distance to the surface of the target structure decreased due to the constantly increasing height of the printed objects.

5. EXPERIMENTAL RESULTS

The StarJet technology features two different dispensing modes, the DropOnDemand-mode and the Continuous-mode (for details see [1]). In the **Continuous-mode** droplets are issued from the nozzle continuously at a certain “natural” frequency. This frequency depends on the nozzle geometry, the applied actuation pressure and other parameters. In several experiments frequencies in the range from $f_{min} = 5$ Hz to $f_{max} = 4$ kHz were observed for different chip designs with nozzle diameters between $50 \mu\text{m}$ and $306 \mu\text{m}$. The chips produced so far enabled to generate droplets with diameters in the range from $d_{drop} = 48 \mu\text{m}$ to $d_{drop} = 360 \mu\text{m}$ dependent on the individual chip geometry. So far two different materials were successfully tested: 1. Solder (Sn95Ag4Cu1) featuring a melting point of $T_m = 210^\circ\text{C}$ and the zinc alloy ZAMAK (Zn96Al4) which melts at $T_m = 420^\circ$. Monodispers single metal droplets could be generated using both materials. Fig.6 shows the dependency of the droplet generation frequency and the droplet diameter on the actuation pressure for chip #12 printing solder. For this chip droplets could be generated at frequencies from 100 Hz to 690 Hz by increasing the actuation pressure in increments of 0.05 bar. Actuation pressures beyond 0.27 bar led to uncontrolled spraying of the metal droplets. At pressures below 0.15 bar the droplet generation stopped.

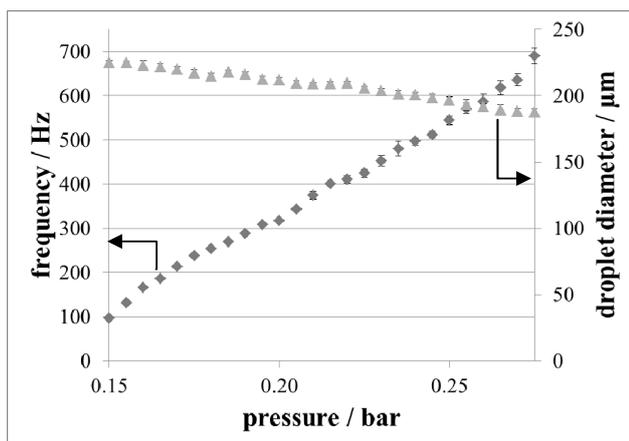


Figure 6: Measurement of break-off frequency and droplet diameter in relation to actuation pressure for chip #12

Each nozzle chip features an individual frequency range depending on the individual nozzle geometry. In our earlier work [7], the droplet diameter was almost

independent on the actuation pressure and for pressures ranging from 0.12 and 0.22 bar it was mainly defined by the nozzle size D_{noz} . With our new setup we detect a 20 % change of the droplet diameters and the droplets get smaller with increasing actuation pressure from 0.15 bar - 0,27 bar. Most probably this is related to the significantly higher frequencies (700 Hz vs. 140 Hz) that we achieve with the improved setup.

The high directional accuracy ($\Delta deg = 0,28^\circ$) of the droplet trajectory allows for stacking single droplets up to a height of 30 mm like demonstrated before [1]. This implies the possibility to produce 3D-structures with high precision by direct printing of metal droplets. The prototyping experiments reported here were performed by dispensing on a rotating table using the setup shown in fig. 5 to create tube – shaped structures. The nozzle chip #6 was applied to generate single droplets of solder (Sn95Ag4Cu1) with a diameter of $d_{drop} = 120 \mu\text{m}$ at a reservoir temperature of $T = 250^\circ\text{C}$ with an actuation pressure of 0.3 bar. The adaptation of the dispensing position, the frequency and the rotation velocity of the substrate allowed for printing homogeneous structures of different diameters ($d = 5,5$ mm to 22 mm) with a typical wall thickness of approximately $s = 300 \mu\text{m}$ (Fig.8).

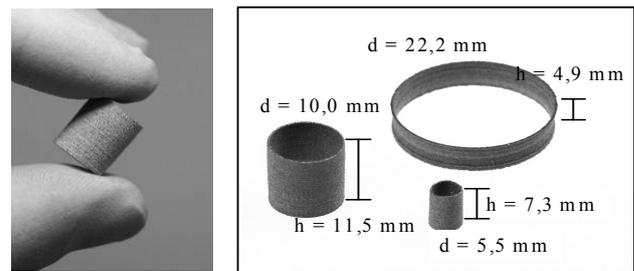


Figure 7: Printed tube-like solder structures

The samples shown in fig. 7 have been created at room temperature without any additional heat conditioning or post treatment. Obviously, the droplets merge and adhere strongly enough to form a reasonably stable, thin walled object. The porosity of the thin wall is clearly visible on the SEM image presented in fig. 8. The close-up view shows that the droplets loose their spherical shape and solidify in a deformed state while impinging on the solid. This leads to the porous structure and a high surface roughness as well as to an increased wall thickness compared to the droplet diameter. The surface of the printed structure has been measured with a profilometer (Tencor, P11) to characterise the roughness of the tube. It turned out that the structure has an average surface roughness of $R_t = 103 \mu\text{m}$. Remarkably there is little oxidization visible on the surface of the printed structures, which is attributed to the pneumatic actuation by Nitrogen as described above. The porosity of the printed structures is desirable for certain applications like the mentioned prototyping of moulding tools for microfluidic applications.

However, often also a good surface finish is required. In principle, liquid surfaces are of optical quality and if

the micro droplets are solidified without splashing very low surface roughness can be obtained. In order to demonstrate this, prototyping experiments with at increased reservoir temperature ($T = 320^{\circ}\text{C}$) have been performed.

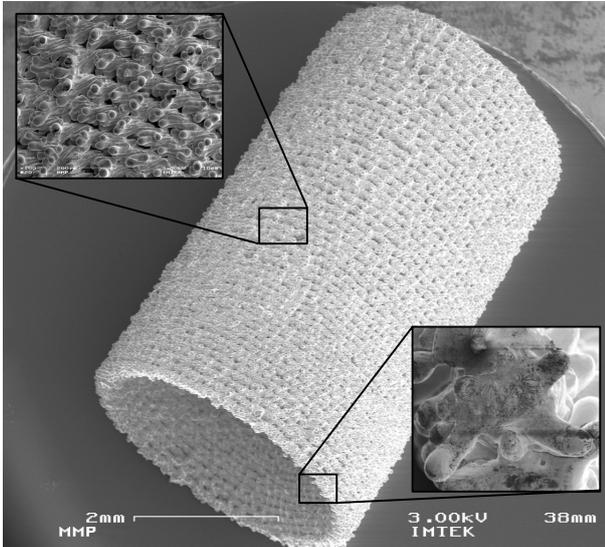


Figure 8: SEM image of a printed micro tube with $315\ \mu\text{m}$ wall thickness. The close-up view shows the morphology of the porous surface.

The higher temperatures of the metal droplets together with an increased dispensing frequencies, caused by a larger actuation pressure ($p_{\text{act}} = 0.45\ \text{bar}$), enabled the droplets to fully merge and to equilibrate, such that the surface tension could create a smooth surface. A sample generated on a substrate at room temperature is shown in fig. 10, entailing a coil-like structure with a smooth surface and without significant porosity. This example demonstrates that it is possible to influence the properties of the printed structures (morphology, porosity, wall thickness, roughness etc.) by variation of the processing parameters.

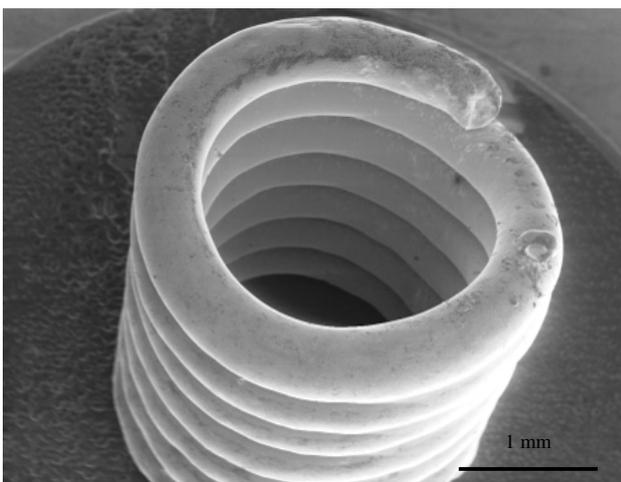


Figure 9: Coil-like structure printed on a heated substrate

6. CONCLUSION & OUTLOOK

The presented results show that the StarJet

technology can be successfully applied for the 3D-prototyping of porous and non-porous metal structures using metals with melting temperatures up to $T = 420^{\circ}\text{C}$. The improved fabrication process of the silicon nozzle-chips increased the quality of the droplet generation in terms of printing frequency ($f_{\text{max}} = 4\ \text{kHz}$) and straightness of the droplet trajectory ($\Delta\text{deg} = 0,28^{\circ}$). The presented tube shaped structures demonstrate that depending on the droplet and substrate temperature, as well as other printing parameters the porosity and wall thickness of the printed structures can be varied considerably.

The presented results are of course only a first step towards the controlled prototyping of metal micro structures by direct printing of liquid metal. More work has to be dedicated to study the solidification and the resulting morphology of the metal surface as a function of materials and process parameters before more complex shapes can be generated. Nevertheless, these first results are encouraging. First of all with respect to the StarJet method that could be possibly applied at even higher temperatures to enlarge the choice of materials to be used for direct metal printing. Furthermore, the simple structures demonstrated in this work could be already suitable for use as micro filters or as micro coils fabricated on chip for RF or micro-NMR applications.

REFERENCES:

- [1] T. Metz, G. Birkle, R. Zengerle, P. Koltay, "StarJet: Pneumatic Dispensing of Nano- to picoliter Droplets of liquid metal" IEEE MEMS 2009, pp.43-46
- [2] M. Focke et al., 2010, Lab Chip Vol.10, pp.2519-2526
- [3] D. Schuhmacher et al. „Erzeugung von Mikrotropfen aus flüssigem Lötzinn mittels einer hochparallelen und kontaktlosen Drucktechnik“ IEEE MEMS 2007, pp. 357-360
- [4] M. ESSION, D.M. Keicher, W.D. Miller, „Manufacturing electronic components in a direct-write process using precision spraying and laser irradiation“, Patent, 2000
- [5] W. Wehl, J.B. Wild, B. Lemmermeyer, „A drop-on-demand metal jet printer for wafer bumping“ Proc. IMAPS 2003
- [6] Wenbin Cao, Miyamoto, Yoshinari, "Freeform fabrication of aluminum parts by direct deposition of molten aluminum" Journal of Materials Processing 173 (2006) 209–212
- [7] A. Tropmann, P. Koltay, N. Paust, R. Zengerle, C. Ziegler : StarJet: Pneumatische Generierung monodisperser Tropfen wässriger Lösungen im Nano- bis Picoliterbereich. SPRAY 2010

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