Extrusion and Deposition of Semi-Solid Metals

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Abstract

Interest in Rapid Prototyping (RP) with fully dense metals is increasing and with it the development of rapid prototyping techniques. A feasibility study for using semi-solid metals as construction materials for rapid prototyping is currently being carried out by TNO and the University of Twente. Semi-solid low melting point alloys are being investigated. Work is being conducted in a number of areas including rheological properties, layerwise deposition and interlayer bonding. An experimental apparatus has been built and tested for the extrusion of these materials. Preliminary results indicate that these materials can be successfully extruded, and the interlayer bonding between layers is satisfactory for the generation of three dimensional objects.

1. Introduction

Rapid Prototyping is becoming an increasingly important part of product development. The rapid production of prototypes enables the manufacturer to adapt his/her design and development process faster to constantly changing requirements.

The Netherlands Organization of Applied Scientific Research (TNO), Institute of Industrial Technology has had considerable success in the development and application of RP techniques, such as Fused Deposition Modeling (FDM), Layered Object Manufacturing (LOM, KIRA) and Multiphase Jet Modeling (MJM) in a wide variety of industries. The FDM technique involves the generation of a near net-shape physical model by extrusion and layered deposition of material based on 3D-CAD files. The systems currently available allow the production of prototypes from thermoplastic materials. However, there is an increasing interest in RP with metals. The use of an FDM-based technique with metals will provide industry with far broader capabilities for product development and a cost effective manner for producing small series of metal products.

The extrusion and deposition of metals, as separate tasks are established production processes. However, for a layered manufacturing technique (LMT) process such as FDM, extrusion and deposition have to be combined in such a manner that the final product fulfils all requirements regarding accuracy and strength. That involves processing the metals in the semi-solid state. In order to control such processes, knowledge about the flow and solidification behavior of semi-solid metals (SSM) is therefore necessary. In particular the design of the equipment for SSM processing requires quantitative data about flow properties, viscosity and solidification behavior. Research is thus being carried out in the fields of semi-solid slurry production, slurry morphology, viscosity, mechanical and metallurgical properties of semi-solids and characteristics of the final products [1].

TNO and the University of Twente have started a co-operative research program to investigate the feasibility of an FDM-based process for the production of 3D metallic objects. Experimental work has begun with low melting point alloys in order to make use of existing testing equipment with minor modifications. Initial work is being conducted to investigate the solidification process of these materials and their flow behavior in the semi-solid state. The preliminary results of this work are presented in this communication.

2. Experimental

Material

For an extrusion process it is fundamental that the material shows a suitable flow behavior. This can be achieved and controlled adequately if the material is being brought into a semi-solid condition. Metals with a wide temperature range where liquid and solid phase coexist are thus required. Properties such as low shrinkage on solidification and no formation of undesired second phase particles have to be considered. The alloys must also have good bonding qualities upon deposition. The alloys shown in Table 1 were found to be suitable with respect to these aspects. These are listed with their respective liquidus and solidus temperatures.

Table 1-Composition and characteristic temperatures of the low melting point alloys

Composition [wt%]	T _{liquidus} [°C]	T _{solidus} [°C]
70%Sn-30%Bi	185	133
35%Sn-35%Pb-30%Bi	140	96
17%Sn-51.5%Pb-31.5%Bi	158	96
60%Pb-40%Sn	235	183

The solidification behavior of these materials was investigated by determining their microstructure after quenching from either the liquid, or the semi-solid state at various cooling rates. The actual goal is to determine the cooling rates during processing based on microstructure since temperature measurement is virtually impossible.

Rheological Data

The flow behavior of the semi-solid materials was investigated by extrusion using a plate/plate rheometer (Fig. 1a) and a capillary viscometer (Fig. 1b). For the latter the material was extruded using several defined ram velocities (see Table 2) and the pressure drop over the capillary was measured. The apparent shear rate can be calculated from the pressure drop as [2]

$$\dot{\gamma}_a = \frac{4Q}{\pi R^3} \tag{1}$$

where Q is the flow rate in the cylindrical part of the capillary and R is the radius. Using the apparent shear rate, the shear rate on the cylinder wall can be calculated according to the Rabinowitsch correction,

$$\dot{\gamma}_{w} = \dot{\gamma}_{a} * \left(\frac{3}{4} + \frac{1}{4} \frac{\partial (\ln Q)}{\partial (\ln \tau_{w})} \right)$$
(2).

In eqn. 2, τ_w is the shear stress at the cylinder wall and is calculated as

$$\tau_w = \frac{\Delta p}{2\frac{L}{R}} \tag{3}$$

where Δp is the pressure drop and L is the length of the capillary. The viscosity can then be calculated as



Figure 1: a) Plate/plate rheometer for the determination of the viscosity as a function of the shear rate and b) Capillary viscometer used for viscosity measurements by extrusion

Table 2- Flow rates for given dimensions and six different ram velocities

Ram velocity [cm/s]	Q [mm ³ /s]	
0.0051	41.2	
0.0103	82.5	
0.0257	206	
0.0515	412	
0.103	825	
0.257	2056	

Initial extrusion trials have been conducted using an extrusion apparatus built similar to the viscometer described above (see Fig 2). There are however, three main differences, the materials, the pressure supply and the nozzle size. The material is supplied in wire form and is transported with a set of 4 friction wheels. The pressure can be varied through the velocity of the friction wheels. In order to create layers of material, the material was extruded through the extrusion head onto a base-plate which could be moved in the x and y directions. Z-axis movement was carried out manually and further layers could thus be deposited.



Figure 2: Extrusion head

3. Results and Discussion

Fig. 3 shows the results of the viscosity measurements using the plate/plate rheometer. The flow curves show a shear thinning behavior which is typical for pseudoplastic materials (Fig. 4). These figures show that the viscosity strongly depends on the shear rate (pseudoplasticity). The shear rate and the time of shearing (thixotropy) are in turn dependent on structural aspects, such as fraction of solid or shape and distribution of particles.



Figure 3: Shear rate vs viscosity measured values



Figure 4: Principal flow curves with a) shear thickening, b) Newtonian and c) shear thinning behavior [3]

The flow curves in Figure 3 and 4 reveal information about the materials reaction to shear forces. Obtaining rheological data on semi-solid systems still remains a problem when it comes to high solid fraction subject to higher shear rates. The torques required are usually too high for conventional viscometers [4]. Extrusion methods in particular have been tried to overcome this problem [5,6]. Fig. 5 shows a typical p-t curve obtained from the extrusion experiments, using the capillary viscometer. After an initial pressure increase the extrusion process starts and the pressure value decreases to a certain level and remains stable. The viscosity at the point where the extrusion starts was determined indirectly using eqns. 1-4 as 884.3 Pa-s.



Figure 5: Pressure-time curve obtained from extrusion tests

Several layers of material have been deposited onto a target using the extrusion device based on these first rheological results. Despite limitations in the experimental setup, it has been possible to deposit not only single layers of metal, but multi-layers as well. The material deposited has been examined using optical and electron microscopy. Fig. 6 shows the areas from which the samples have been prepared and Fig. 7 the microstructure of a) extruded material and b) an interlayer area. Microstructures of all extruded specimens appeared globular, which is typical for semi-solid metal, having been subjected to shear forces. Quenching tests showed that this microstructure can be related to a cooling rate of approx. 6°C/s. Clear bonding between the two layers can be observed. The bonding could be further improved if a protective atmosphere and elevated chamber temperatures are introduced in the process.







Figure 7: Micrograph showing a) the microstructure of extruded material and b) an interlayer region

The work conducted thus far has shown that the extrusion and deposition of metallic materials is feasible for the creation of physical 3D objects. Further work will concentrate on the deposition process. The x-y-z-movement will be fully automated so that controlled deposition is possible. The relationship between the extrusion velocity and x-y movement are critical to the process and require optimization. This also applies to the start-stop control of the materials flow. Rheological models will be employed in order to optimize the nozzle design and thus improve the extrusion process.

The mechanical properties of the extruded material will be tested. Preliminary tests showed that under certain circumstances the bismuth containing alloys became brittle. SEM analysis of these materials revealed that a bismuth-rich phase in the form of coarse particles formed at grain boundaries and caused embrittlement. This problem could be solved through a variation of process parameters or materials composition.

4. Conclusions

A program is being conducted to show the feasibility of Rapid Prototyping with metals using an FDM-based technique. Initial work with Sn-based low melting point alloys has shown that layer deposition of these materials is possible by extrusion in the semi-solid state. Further studies on the relationship between microstructural and rheological properties of these metals are being conducted in order to optimize the equipment and process.

5. References

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