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Paper:

Shape Contraction in Sintering of 3D Objects Fabricated via Metal Material Extrusion in Additive Manufacturing

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Additive manufacturing (AM) using metal materials (metal AM) is useful in the fabrication of metal parts with complex shapes, which are difficult to manufacture via subtractive processing. Metal AM is employed in the manufacture of final products as well as in prototyping. Recently, certain metal-AM machines have been commercialized. Powder-bed fusion and direct energy deposition are the main types of metal AM; they require the use of a high-power laser or electron beam and most of them are highly expensive. On the other hand, AM machines of the material-extrusion (ME) type can fabricate metal parts at a low cost. ME is the method of extruding materials from a nozzle and fabricating thin layers. By mixing a metal filler with a base material, it is possible to impart various mechanical properties to the extruded material, such as electrical or thermal conductivity. If the extruded material is baked in a furnace after fabrication, the object can be sintered. During the sintering process, the fabricated objects always shrink and dimensional errors occur. One of the reasons for the shrinkage is that voids are generated inside the object after the degreasing process and collapse during the sintering process. Because the void is generated as a space by replacing a binder that becomes vaporized during the degreasing process, the shrinkage may be controlled by decreasing the content in polymers. In this study, the effect of the metal filler density on the shrinkage in shape was investigated through experiments using two types of metal ME AM. One type is the fused filament fabrication (FFF), in which a material that consists of a metal filler and fused plastics is extruded; the other type is the ultrasonic vibration-assisted ME (UVAME) device, in which a metal powder suspension with a small amount of thickening polymer is extruded. In the latter method, materials with an extremely high density in metal fillers were used; it was considered that degreasing was not required. Two types of specimens were fabricated using AM devices; they were then degreased and sintered. The resulting shapes of the objects were measured with a 3D scanner and were compared. The experimental results showed that the shrinkage of the material with a high density of metal fillers was less than that of the material with a low density of metal fillers.

Keywords: additive manufacturing, metal material, material extrusion, sintering, shrinkage

1. Introduction

Additive manufacturing using metal materials (metal AM) is useful in the fabrication of metal parts with complex shapes, which are difficult to be manufactured via subtractive processing. Recently, metal AM has been employed in the manufacturing of final products in various fields, such as in aviation [1] or in the medical field [2]. Moreover, studies have been conducted on the functionality of mold fabrication through metal AM [3, 4].

The main processing types of metal AM are powder-bed fusion and direct energy deposition. Several studies have been conducted on the strength of the fabricated objects or the fabrication conditions of the aforementioned processing types [5–7]. More specifically, studies on the strength or the fracture characteristics of functional structures, such as cellular structures [8, 9] or porous structures [10, 11], fabricated via the aforementioned processing types were conducted. However, these metal AM machines require a high-power laser or electron beam and they are currently highly expensive. Therefore, it is difficult for them to be used in small shops.

The less expensive processing types of metal AM are the material-extrusion (ME) type and the binder-jetting type AM machines. In ME-type AM machines, the fused filament fabrication (FFF) – in which melted materials are extruded from the machine nozzle – is a popular method. However, the melting point of metals is very high and the direct extrusion of melted material is difficult. Therefore, as a solution, materials containing metal fillers are extruded from the machine nozzle and undergo a sintering process. In the present work, this method will be referred to as metal ME. In general, fillers are often mixed into polymers to improve mechanical [12], thermal [13], or electrical [14] properties. Moreover, there are several studies on composite-material filaments for the fabrication of medical [15] or multi-functional materials [16].

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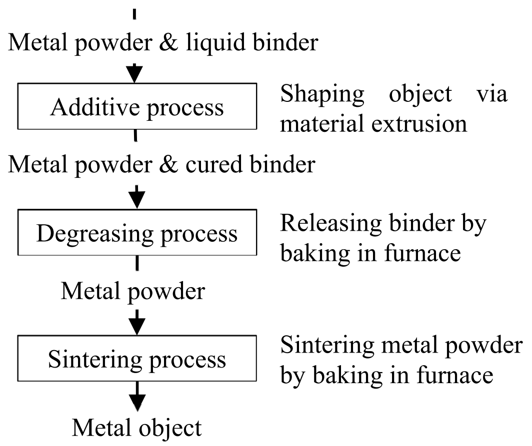


Fig. 1. Fabrication process of metal parts by using metal powder in ME-type AM.

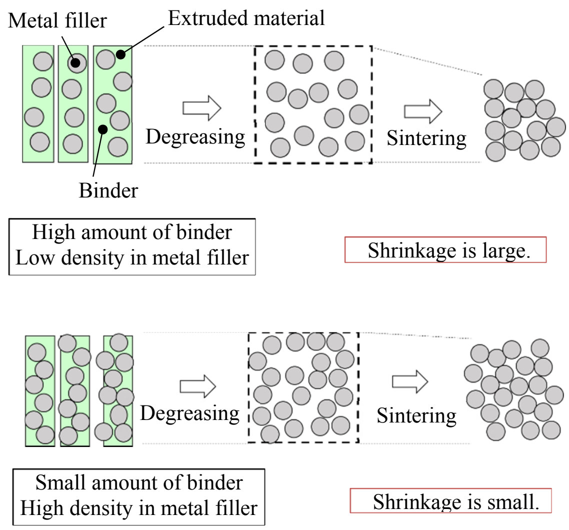


Fig. 2. The shrinking process during the thermal process.

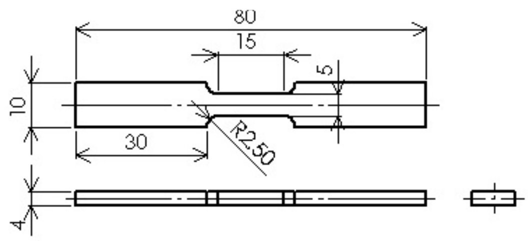


Fig. 3. Drawing of the specimen.

In metal ME, materials with metal fillers contain a high amount of resin as a binder to ensure stable extrusion and to maintain the shapes of the objects after the extrusion. After the degreasing process, in which the binder is removed, the sintering process follows. When the fabricated objects are sintered, they always shrink and present dimensional errors.

The binder-jetting type of AM using metal powder has also been recently developed. There are certain studies on the density change or the shrinkage of metal parts [17] or ceramic composites [18] fabricated via binder-jetting type AM machines. However, in this method, the object requires sintering after its fabrication as well.

Therefore, in the present study, the specimens were fabricated with two types of metal-ME machines. One type was the FFF machine. The fused filament extruded using the FFF machine contains metal fillers. The other was the ultrasonic vibration-assisted ME (UVAME) device, which extrudes metal powder suspension [17, 19]. In this method, materials with an extremely high density of metal fillers can be used. Degreasing is not required because the amount of binder is small. After these two types of specimens had been fabricated and sintered, the shape of the object was measured with a 3D scanner and the dimensional errors were calculated.

2. Fabrication Process

Figure 1 shows a general fabrication process through material extrusion using metal powder. First, materials for AM are prepared by mixing metal powder and binder. Then, the material is extruded with a planar motion in order to be placed in an appropriate position. In the material extrusion process, the binder is used in a liquid state. The material is deposited layer by layer, which is the additive process. Next, the shaped object is baked in a furnace to release the binder from the object, which constitutes the degreasing process. Then, the object is baked again at a high temperature for sintering; this is the sintering process. Finally, a metal object is obtained.

The assumed material behavior during the thermal process is shown in **Fig. 2**. Initially, the extruded material containing the metal powder and binder is shaped as a line or curve with a gap between adjacent extrusions because each of them is extruded separately. In the degreasing and sintering process, the binders that help the objects maintain their shape are removed. There is a proportional relation between the binder content in the extruded materials and the shrinkage of sintered objects.

3. Experimental Device and Materials

3.1. Shape and Material of Specimen

Figure 3 illustrates the mechanical drawing of a specimen. The specimens were fabricated with a commercial FFF-type AM machine (Axiom Dual, Airwolf 3D Printing), which is shown in **Fig. 4**; they were made of a metal-polymer composite filament (Ultrafuse 316LX, BASF), shown in **Fig. 5**. **Table 1** lists the fabrication conditions set for the FFF-type AM machine. In addition, to compare the properties, the specimens were fabricated using a UVAME device, which extrudes metal powder suspension. In this method, materials with extremely high density of metal fillers can be used. **Fig. 6** shows the UVAME device. In this method, the ultrasonic vibration materi-

Fig. 4. FFF-type AM machine.

Fig. 5. Metal-polymer composite filament.

Table 1. Fabrication condition of the FFF-type AM machine.

Nozzle diameter	0.4 mm
Nozzle temperature	240°C
Travel speed	35 mm/s
Layer thickness	0.22 mm

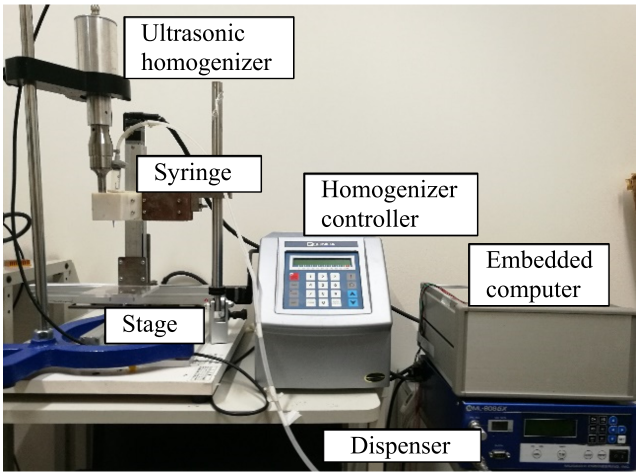


Fig. 6. UVAME apparatus.

als can be extruded smoothly. Thixotropic materials are used for maintaining the shape of the objects after extrusion [17, 19]. Thixotropy is the property in which viscosity decreases when vibration is applied, and it is recovered when the vibration is removed. In this experiment, the apparent viscosity of the metal powder suspension decreased as it vibrated with a homogenizer. Then, at air pressure, the material was extruded smoothly. A water-solvable polymer powder (AEROSIL 200, EVONIK) was used as the thixotropic material and was mixed with stainless steel powder (LPW-316-AAAV, LPW) at the mass ratio listed in **Table 2**. **Table 3** summarizes the fabrication conditions.

3.2. Degreasing and Sintering

The specimens fabricated with the FFF machine were degreased in the electric furnace (FT-01VAC-1650, Full-Tech) at 350°C in a vacuum environment. In addition, the sintering process was realized in the electric furnace in a vacuum environment as well. The sintering temperature and time were based on past research on metal injection molding (MIM) [20]. The sintering conditions are listed in **Table 4**. In addition, the specimens were fabricated using the UVAME device and were sintered as well.

3.3. Measuring Dimensions

The shape of the specimens was measured with a 3D scanner (NextEngine, 3D Systems Japan). The resolution was 100 μm. The specimen dimensions are illustrated in **Fig. 7**; they were obtained using a 3D-CAD software (RapidWorks, 3D Systems), along with the volume of the specimens. The 3D data of the shape of the specimen were processed with 3D-CAD software and were converted into polygon data, from which the volume of the test piece could be calculated.

Table 2. Material mass ratio of the metal powder suspension.

Materials	Mass [g]
Steel	45
Polymer	1.0
Water	3.5

Table 3. Fabrication conditions of the ultrasonic vibration-assisted metal ME device.

Nozzle diameter	0.4 mm
Travel speed	4 mm/s
Vibration frequency	20 kHz
Vibration output power	100 W
Pressure	100 kPa

Table 4. Sintering conditions.

Step	Condition	Target temperature	Running time
1	Raising	1350°C	30 min
2	Holding	1350°C	2 h
3	Cooling	200°C	2 h

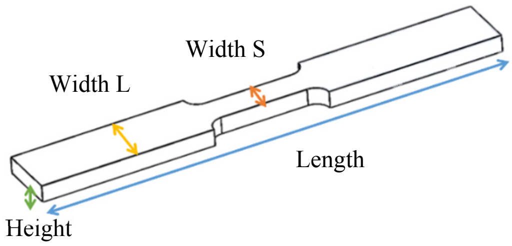
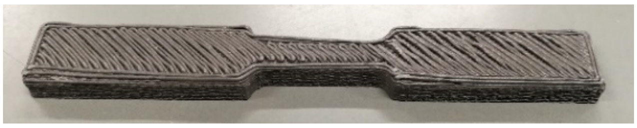


Fig. 7. Measuring points.

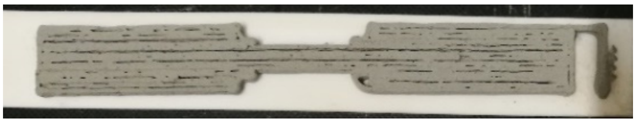


(a) After the additive process.

(b) After the degreasing process.

(c) After sintering process.

Fig. 8. A specimen fabricated using the FFF-type AM machine.



(a) After the additive process.

(b) After the sintering process (back side).

(c) Metallic luster after polishing.

Fig. 9. A specimen fabricated using the UVAME device.

4. Experimental Results and Discussion

4.1. Fabrication, Degreasing, and Sintering

Two specimens, referred to as FFF-1 and FFF-2, were fabricated using the FFF-type AM machine. **Fig. 8** shows images of the FFF specimens after the additive (**Fig. 8(a)**), the degreasing (**Fig. 8(b)**), and the sintering (**Fig. 8(c)**) processes. Two specimens, referred to as UVAME-1 and UVAME-2, were fabricated using the UVAME machine. **Fig. 9** shows images of the UVAME specimens after the additive (**Fig. 9(a)**) and the sintering (**Fig. 9(b)**) processes. By polishing the specimen with a file, metallic luster was revealed, as shown in **Fig. 9(c)**.

Table 5 lists the dimension errors in the four types of dimensions that are shown in **Fig. 7** after fabrication. As may be observed, relatively significant dimensional errors occur in the height of UVAME-1 and the width, Width *S*, of UVAME-2. These specimens were fabricated with the UVAME device. We assume that the extrusion decreased because the nozzle was clogged.

Table 5. Dimensional errors of the fabricated specimens.

	Dimensional errors [mm]			
Specimen	FFF-1	FFF-2	UVAME-1	UVAME-2
Length	−0.5	0	0.4	1.6
Height	0.33	0.2	−1.16	−0.13
Width *S*	0.42	0.42	0.28	−1.28
Width *L*	0.8	0.4	−0.28	−0.31

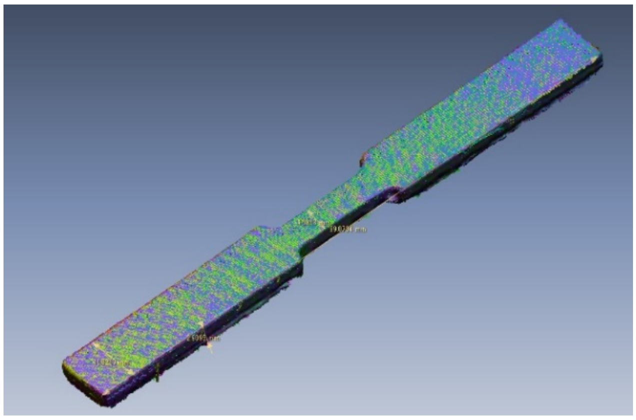


Fig. 10. An example of shapes scanned with the 3D scanner.

4.2. Shrinkage

Figure 10 shows an example of the shapes scanned with the 3D scanner. **Fig. 11** illustrates the shrinkage of the specimens fabricated using the two types of metal AM.

In **Fig. 11**, it may be observed that the specimens fabricated using the FFF-type AM machine shrink more than the ones fabricated using the UVAME device after sintering.

In **Figs. 11(a)** and **(b)**, the width of the specimens decreases by approximately 20% and the height decreases by approximately 30%. However, the width of the specimens after the degreasing process increases.

On the other hand, a common characteristic that may be observed in **Figs. 11(c)** and **(d)** is that the height after the sintering process increases. This occurred owing to the uniformity of the layer height of the UVAME objects. The material in the FFF-type AM machine was cured instantly after the extrusion process. Therefore, the layer pitch was constant. On the other hand, the viscosity of the metal powder suspension in the UVAME recovered more slowly than that in the FFF. Therefore, in the UVAME, the lower layers were pushed down and became flat because of the weight of the upper layers. Additionally, the shape of the object resembled a mountain. Moreover, it was found that the volume of the materials that had been extruded from the nozzle gradually minimized after a long extrusion process [19]. **Fig. 12** shows the shrinkage process during the sintering process. The outer part of the lower

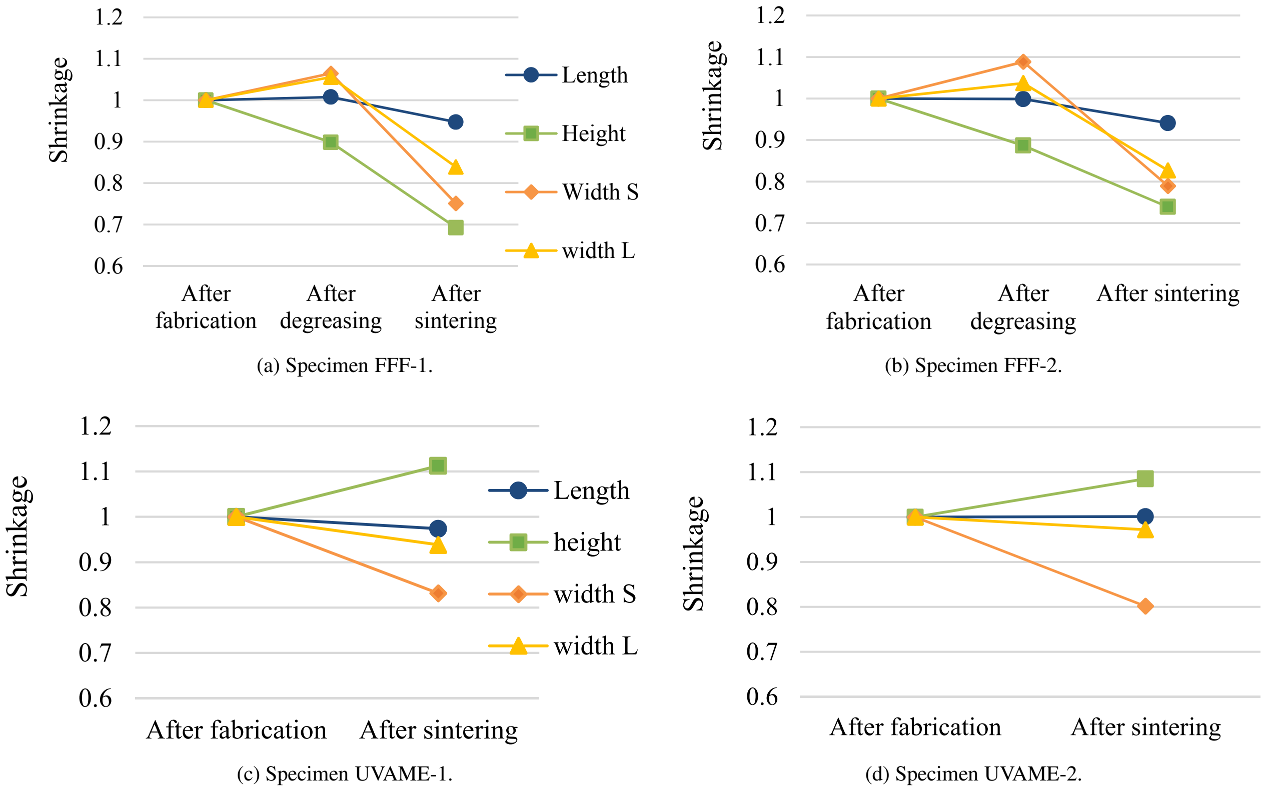


Fig. 11. Shrinkage during the thermal processing of specimens via metal AM.

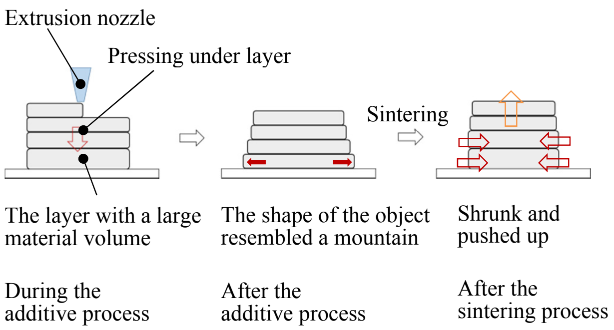


Fig. 12. The shrinkage process of objects fabricated with the UVAME device.

layers shrinks faster than the inner part, which becomes tighter and is pushed toward the upper layers. Therefore, the height after the sintering process increases.

In addition, the volume shrinkage of the specimens fabricated using the two ME types of metal AM are listed in **Table 6**. FFF-1 and FFF-2 shrunk by approximately 30% of the volume. On the other hand, UVAME-1 and UVAME-2 shrunk by a smaller percentage.

5. Tensile Test

A tensile test was conducted to identify the qualitative properties of the sintered specimen fabricated using the FFF-type AM machine. A universal testing machine (Shimazu Corporation, EZ-LX) was employed. The specimens were subjected to tension until they broke at a speed of 2.0 mm/min.

Table 6. Volume shrinkage.

Specimen	Shrinkage volume
FFF-1	28.2%
FFF-2	27.5%
UVAME-1	−1.14%
UVAME-2	−2.69%

The tensile stress was calculated using the tensile load that was measured with the testing machine; the cross-sectional area was measured with a 3D scanner. This tensile test was conducted to identify the qualitative characteristics of the materials. **Fig. 13** shows the stress – strain curve of FFF-1. **Fig. 14** shows the fractured surface of the specimen. The tensile strength was 82.9 MPa. The specimen broke soon after yielding. This result reveals that the ductility of the material was lower than that of steel. The value of Young's modulus was small as well. Several air gaps were observed in the fractured surface; therefore, cracks may be easily generated. Because it is difficult to fabricate objects without air gaps using the ME-type AM machine, it is not suitable for machined parts that require high strength or rigidity. However, the productivity of the material extrusion is higher than that of the powder-bed-

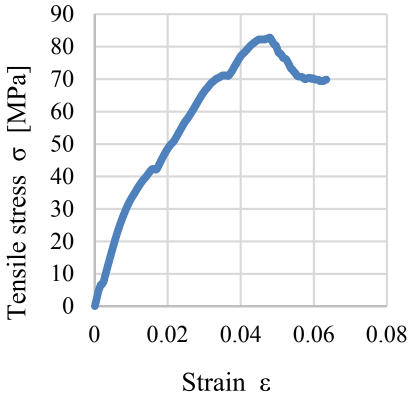


Fig. 13. Stress-strain curve.

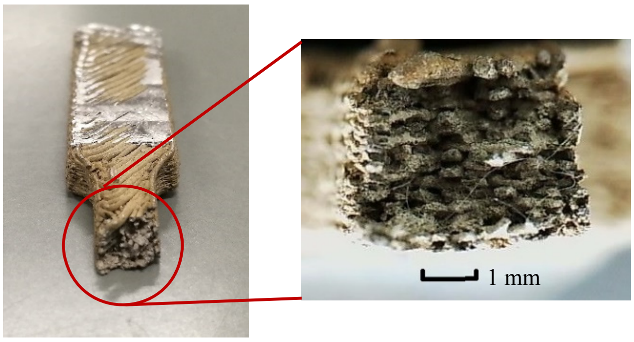


Fig. 14. Fractured surface of the specimen fabricated using the FFF-type AM machine.

fusion-type AM, and the strength of the material is higher than that of the resin. Therefore, the UVAME machine is suitable for the fabrication of large parts that require suitable strength and rigidity or that have free shapes, including their inner structure.

6. Conclusion

(1) By using two types of metal ME AM machines, specimens were shaped via the additive process and were baked via the sintering process.

(2) The specimens before and after the sintering process were scanned with a 3D scanner to obtain their shape and the shrinkage was measured.

(3) It was confirmed that there is a relation between the volume of the binders in extruded materials and the shrinkage of the sintered objects.

(4) A tensile test was conducted on the specimen fabricated using the FFF-type metal AM. The results showed that the sintering process was performed effectively.

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