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Multiphase Polymeric Materials for Rapid Prototyping and Tooling Technologies and Their Applications

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Abstract

Rapid prototyping (RP) and tooling (RT) are the technologies for quickly fabricating functional components and tooling inserts directly from CAD data by selectively adding material layer by layer. In this paper, multiphase polymeric materials for RP and RT technologies and their applications, which are developed by the Rapid Manufacturing (RM) Center of Huazhong University of Science and Technology (HUST) in China, were introduced. Selective laser sintering (SLS) is a powder-based RP process. Multi-types of multiphase polymer materials for SLS process were successfully developed in the RM center, and the SLS components were formed from these materials by using the commercial SLS machines HRPS series for various applications. High impact polystyrene (HIPS)/wax blend SLS parts were used as lost patterns for the investment casting process to make complex metal parts rapidly; nylon-12/organically modified rectorite and nylon-12/nanosilica composite powders were used to fabricate functional parts, which showed higher thermal and mechanical properties than neat nylon-12 SLS parts. As a RT application, Fe/epoxy composite tooling inserts were rapidly fabricated by SLS and post-processing. Stereolithography (SLA) uses photo-curable resins to rapidly manufacture components with high accuracy and mechanical properties. A free-radical and cationic mixed-type radiation curable composite resin was also successfully developed, and SLA parts without obvious distortion were built on the SLA machines HRPL series from this hybrid resin, successfully and efficiently.

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Keywords

Rapid prototyping, rapid tooling, multiphase polymeric materials

1. Introduction

Rapid prototyping (RP) technologies, such as selective laser sintering (SLS), stereolithography (SLA), fused deposition modeling (FDM) and laminated object manufacturing (LOM), are capable of quickly producing complex freeform solid objects directly from a computer model of an object without the need for tooling. RP tech-

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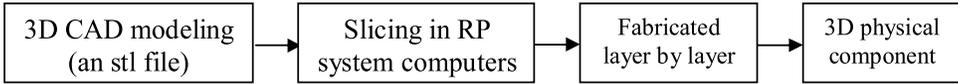


Figure 1. Flow diagram of the rapid prototyping (RP) process.

nologies are additive processes. RP components are built up gradually in layers until the final geometry is obtained. The way in which the layers are produced, however, and the materials in which parts can be built vary significantly amongst the different RP technologies. The RP process is illustrated in Fig. 1. The starting point for the RP process is typically a 3-D CAD model prepared and exported to meet the requirements of a given technology. The original data is reformatted into an STL file and sliced horizontally, and each individual slice is subsequently presented to the selected RP manufacturing process. The RP system will reproduce the slices data thereby creating a physical example of the original ‘CAD’ data [1–4].

Rapid tooling (RT) technology embodies the creation of prototype or production tooling inserts based on RP parts. Using the tooling inserts made by RT technology, large quantities of similar parts containing complex features can be made economically from materials close to or identical to end production materials [5–8].

Nearly all commercial RP technologies currently produce parts from polymeric or oligomeric hydrocarbon materials. The dominance of polymeric materials, relative to metals and ceramics, is largely a consequence of their ease of processing at relatively modest temperatures, generally less than 400°C. This permits a lower-cost energy source to be employed by the shaping or fabricating technology than are required to directly fabricate with metals and ceramics. In addition, polymeric materials have low surface energies, relative to metals. This feature eliminated the tendency seen in the RP processes of metals for the molten material to minimize its energy by forming spherical droplets that can cause poor surface quality [3]. Polymeric materials have an especial advantage, relative to metals and ceramics. Their mechanical, thermal and rheological properties can be easily modified and optimized by forming multiphase polymeric systems such as blends and composites for each specific RP technology to make parts for the different applications. In this paper, multiphase polymer materials for RP and RT technologies and their applications, which are developed by the Rapid Manufacturing (RM) Center of Huazhong University of Science and Technology (HUST) in China, were introduced.

2. Selective Laser Sintering

SLS is an advanced RP technology, which applies a laser beam to create three-dimensional objects from powdered materials. In the SLS process, illustrated schematically in Fig. 2, a very thin layer of heat-fusible powdered material is delivered onto the movable platform by a roller. A heat-generating CO₂ laser beam traces across this layer, sintering specific areas according to the instructions of the computer aided design (CAD) model. The platform lowers slightly and another thin

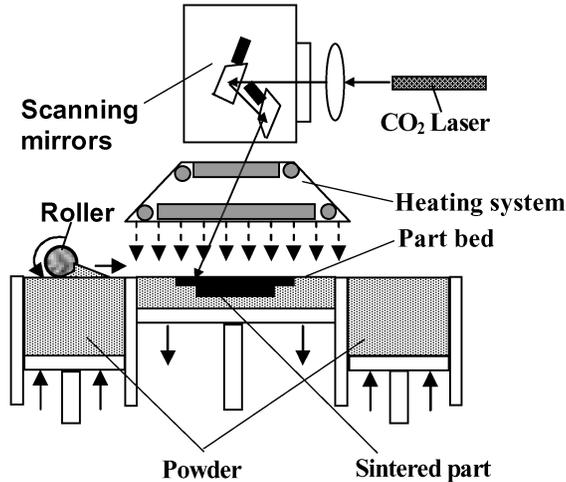


Figure 2. Schematic illustration of the SLS process.

layer of material is delivered. Then, the laser beam scans selected areas of this layer which bonds to the previous layer. The process continues, layer by layer. The sintered material forms the object, while the unsintered powder remains in place to support the structure and is cleaned away and recycled once the build is completed [9–12].

The SLS process can be used on amorphous or semi-crystalline polymer powders. Semi-crystalline polymers, such as nylon-12 [13–22], nylon-1010 [23], nylon-6 [24] and nylon-11 [25, 26], have been used to produce functional parts by SLS for their nearly fully dense structure and good mechanical properties. Amorphous polymers, such as polycarbonate (PC) [27–33], polystyrene (PS) [34–36] and high impact polystyrene (HIPS) [37, 38] have been widely used as SLS materials. Normally, the SLS parts of amorphous polymers are porous, and can be reinforced by infiltrating with epoxies and other thermoset resins for the applications of functional parts.

Multi-types of multiphase polymeric materials for SLS process were successfully developed in the RM center of HUST, and the SLS components were formed from these materials by using the commercial selective laser sintering machine HRPS series, and widely applied in many fields.

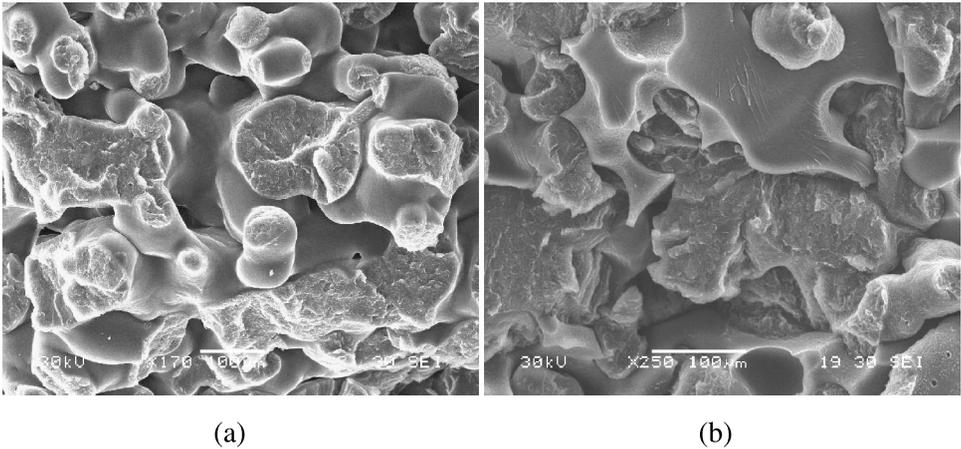
2.1. High Impact Polystyrene/Epoxy Resin Blend

High impact polystyrene (HIPS) is used as an SLS material [37]. Because HIPS is an amorphous polymer, its SLS parts are porous, possessing high dimensional accuracy and relatively low mechanical properties. Therefore, the original HIPS SLS parts are reinforced by infiltrating with epoxy resin. Table 1 lists the mechanical properties of the HIPS SLS specimens before and after infiltrating with epoxy resin. From Table 1, it can be found that the mechanical properties are greatly enhanced after infiltrating with epoxy resin. Figure 3(a) and 3(b) shows the SEM

Table 1.

Mechanical properties of HIPS and HIPS/epoxy SLS specimen

Materials	Tensile strength (MPa)	Ultimate elongation (%)	Flexural strength (MPa)	Impact strength (kJ/m ²)
HIPS	4.59	5.79	18.93	3.30
HIPS/epoxy	18.63	6.44	30.69	3.90

**Figure 3.** SEM photographs of the tensile fractured surfaces of the HIPS SLS specimens (a) before and (b) after infiltrating with epoxy resin.

photographs of the tensile fractured surfaces of the HIPS SLS specimens before and after infiltrating with epoxy resin, respectively. It can be seen that before infiltrating, there are a plenty of voids inside the HIPS SLS parts. After infiltrating with epoxy resin, nearly all voids are filled with epoxy resin, and interfacial adhesion between the two materials is good. Consequently, the HIPS/epoxy SLS specimens have much higher mechanical properties than the HIPS SLS specimens and can be used as functional parts. Figure 4 shows an HIPS/epoxy SLS part with complicated structures.

2.2. High Impact Polystyrene/Wax Blend

The HIPS SLS parts can also be reinforced by infiltrating with wax, and obtained HIPS/wax SLS parts are used for the investment casting technology to make metal parts [38]. Infiltrating with wax has the following three functions.

- (1) It improves the mechanical properties of the SLS green parts and provides sufficient strengths for the post-processing. Table 2 lists the mechanical properties of the HIPS SLS parts before and after infiltrating with wax. From the data of



Figure 4. A HIPS/epoxy SLS part with complicated structures.

Table 2.

Mechanical properties of the HIPS SLS parts before and after infiltrating with wax

Specimens	Tensile strength (MPa)	Ultimate elongation (%)	Flexural strength (MPa)	Impact strength (kJ/m ²)
Before infiltrating	4.59	5.79	18.93	3.30
After infiltrating	7.54	5.98	20.48	6.50

Table 2, it is found that the tensile strength, elongation, flexural strength and impact strength are all increased after the green parts are infiltrated with wax.

- (2) As the SLS HIPS parts are porous, they must be sealed prior to dipping in ceramic slurry for casting. Through the infiltration with wax, the green parts are sealed.
- (3) The green parts have rough surfaces. By infiltrating with wax, the surface roughness of the green parts can be decreased.

After the HIPS/wax SLS part is fabricated, the part is dipped in a ceramic slurry to make a ceramic molding shell. When the part coated with a layer of ceramic is dried, it is baked in a vacuum oven to remove the resin and wax, and then a moulding shell for casting is finished. The last step is to pour molten metal into the moulding shell, and then a metal part is made. Figure 5(a) shows a HIPS/wax SLS part with complicated structures. The metal part obtained from the HIPS/wax part by investment casting technology is shown in Fig. 5(b).

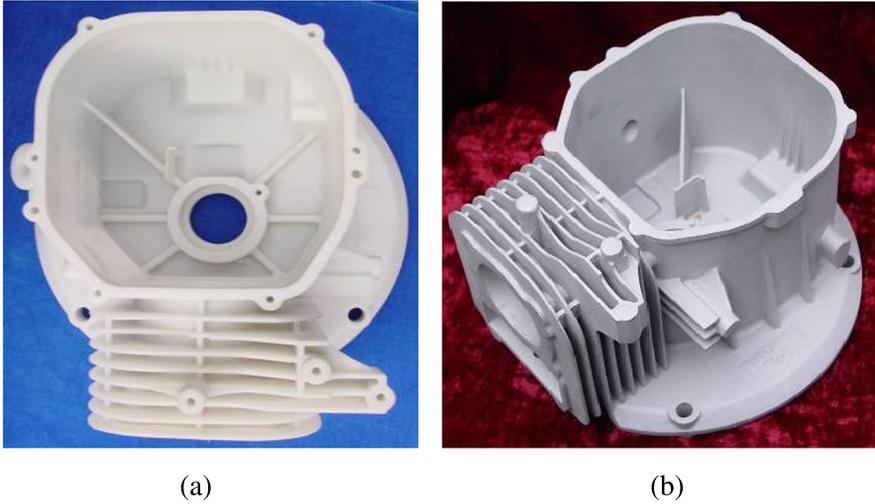


Figure 5. (a) A HIPS/wax SLS part with complicated structures. (b) Metal part obtained from the HIPS/wax part by investment casting.

2.3. *Polystyrene/Polyamide Alloy*

Polystyrene (PS) powder materials may be directly used for selective laser sintering (SLS). The part sintered with PS has high dimensional accuracy, but its tensile strength is very low (only 5–6 MPa) and its solvent resistance capability is not good, which cannot meet the demands of the functional parts. The part sintered with polyamide (PA) has high mechanical properties and excellent resistance capability, so PA is suitable for building functional parts. However, the dimensional accuracy of the SLS part sintered with PA is low. Also, PA's price is very high (relative to PS powder). Colligating the properties of the two materials, PS and PA, a polymer alloy of PS and PA for building functional parts by SLS process is developed [36]. In the PS/PA alloy system, PS-g-MAH (maleic anhydride graft co-polymer) is used as the compatilizer. When the PS powder is blended with the PA powder, the anhydride-radical of the compatilizer has a reaction with the amido of the PA's macromolecular chain. The compatibility of PA and PS is improved by the 'bridge function' of the compatilizer, and the alloy tensile strength is enhanced. The polymer alloy whose formulation is PS/PA/SMA = 80/20/5 (mass ratio) has better compatibility. The alloy materials have better sintering properties, and the SLS parts fabricated with the alloy materials have a higher tensile strength of 14.4 MPa, and better solvent resistant capability. The alloy materials can meet the fabrication demands of the functional part.

2.4. *Organically Modified Rectorite/Polyamide-12 Nanocomposites*

Organically modified rectorite (OREC) is blended with polyamide-12 (PA12), then sintered by laser to fabricate specimens [17]. Figure 6 shows the XRD patterns of both the OREC and the PA12–OREC (5 wt%) sintered composite. Figure

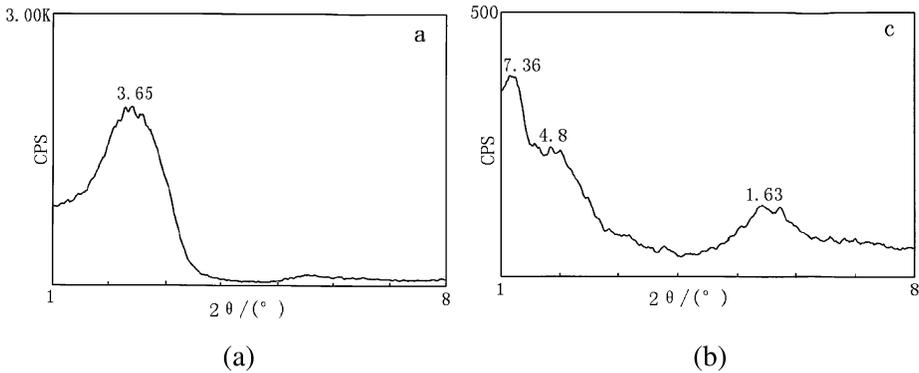


Figure 6. XRD patterns of (a) OREC and (b) PA12/OREC (5 wt%).

Table 3.

Mechanical properties of the PA12 and PA12/OREC (5 wt%) SLS specimens

Materials	Tensile strength (MPa)	Ultimate elongation (%)	Flexural strength (MPa)	Impact strength (kJ/m ²)
PA12	38.3	20.83	50.3	30.4
PA12/OREC (5 wt%)	50.9	18.2	67.3	33.8

6(a) and 6(b) shows the XRD patterns of OREC and PA12/OREC (5 wt%), respectively. The XRD pattern of the OREC (see Fig. 6(a)) shows a characteristic peak (d001) around 3.65 nm. From Fig. 6(b), it can be found that after mixing and sintering with PA12, the diffraction peak corresponding to the OREC is 7.36 nm. This result implies that OREC was intercalated by PA12 during the sintering process and intercalated PA12/OREC nanocomposites were formed.

The mechanical properties of the PA12 and PA12/OREC (5 wt%) SLS specimens are listed in Table 3. From Table 3, the tensile strength, flexural strength and impact strength of the PA12/OREC (5 wt%) SLS specimens are all higher than those of the PA12 SLS specimens. From the above results, it can be concluded that OREC has a reinforcement effect on PA12 SLS parts. Figure 7 shows SLS parts of PA12/OREC (5 wt%) with complicated structures.

2.5. Nanosilica/Polyamide-12 Composite Powder

A dissolution–precipitation process was successfully developed to prepare a nanosilica/PA12 composite powder for the SLS process [18]. Nanosilica modified by a silane coupling agent was first dispersed uniformly in the nylon-12 alcohol solution by ultrasonic oscillation and stirring constantly at about 160°C. Then, the mixture was cooled gradually, PA12 crystallized taking the nanosilica particles as heterogeneous nuclei and the composite powder was formed gradually. The dispersion of nanosilica in the SLS parts of the composite powder was examined by field

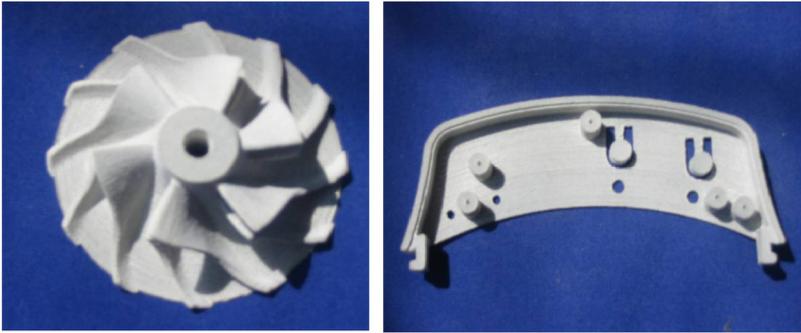


Figure 7. SLS parts of PA12/OREC (5 wt%) with complicated structures.

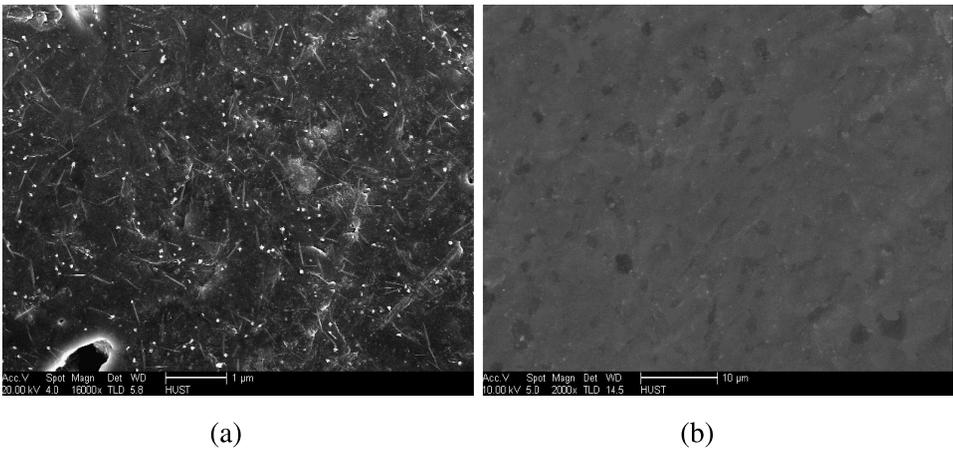


Figure 8. SEM photographs of the cryogenically fractured surfaces of the (a) nanosilica/PA12 and (b) PA12 SLS specimens.

Table 4.

Mechanical properties of the SLS specimens made from the nanosilica/PA12 and PA12 powders

Samples	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)	Impact strength (kJ/m ²)
Nanosilica/PA12	46.3	20.07	1.98	40.2
PA12	38.3	20.83	1.42	30.4

emission scanning electron microscope. Figure 8(a) and 8(b) shows the SEM photographs of the cryogenically fractured surfaces of the nanosilica/PA12 and PA12 SLS specimens, respectively. From Fig. 8(a), it can be observed that nanosilica particles are uniformly dispersed on a nano-scale level in the SLS specimens.

The mechanical properties of the SLS specimens made from the nanosilica/PA12 and PA12 powders are listed in Table 4. From Table 4, it can be found that the tensile

Table 5.
Mechanical properties of Fe/epoxy composites for RT

Samples	Flexural strength (MPa)	Flexural modulus (GPa)
Fe/epoxy composites	102.3	14.2

strength, modulus and impact strength of the nanosilica/PA12 SLS specimens are all higher than those of PA12, indicating that nanosilica has a good reinforcement effect on the PA12 SLS parts.

3. RT Material

Fe/epoxy resin composite tooling inserts had been successfully fabricated by SLS and some post-processes in the RM center of HUST. The manufacturing procedure is as following. A nylon-12 coated Fe powder is prepared by the dissolution–precipitation process. The green parts of tooling inserts with internal conformal cooling channels are manufactured with this coated Fe powder by the SLS process. Then the polymer binder of nylon-12 in the green parts is decomposed at about 500°C. The green parts are subjected to high temperature sintering at 1200°C for 10 h. Finally, the tooling inserts parts are infiltrated with an epoxy resin, and cured at 100°C for 10 h, then 160°C for 5 h. The mechanical properties of the Fe/epoxy composites are listed in Table 5. The obtained tooling inserts made of Fe/epoxy resin composites can be used for plastic injection. Figure 9(a) shows the tooling inserts parts after infiltrating with the epoxy resin. Figure 9(b) shows the tooling inserts made of Fe/epoxy resin composites after machining to improve surface roughness. The plastic parts from the injection of the tooling inserts are shown in Fig. 9(c). The characteristics of the tooling inserts made of Fe/epoxy composites are as follows. (1) The time required to finish a typical core and cavity mold set is at about 7 days; (2) A finished mold can be used to produce in excess of 500 parts; (3) Complicated conformal cooling channels can be manufactured by the SLS process.

4. Stereolithography

SLA is one of the oldest RP technologies dating back to the mid-1980s. SLA can be used to make parts with complex geometry and with a surface finish comparable to many conventionally machined components. Figure 10 schematically illustrates the SLA process. A vat of polymer contains a platform on which the part is built. The platform can rise and fall within the vat. The platform moves until it is just below (0.050–0.250 mm) the surface of the liquid polymer. An UV laser beam traces out the cross-section of one slice of the part. Where the UV laser beam hits the polymer it solidifies. The platform then moves down the distance of one slice

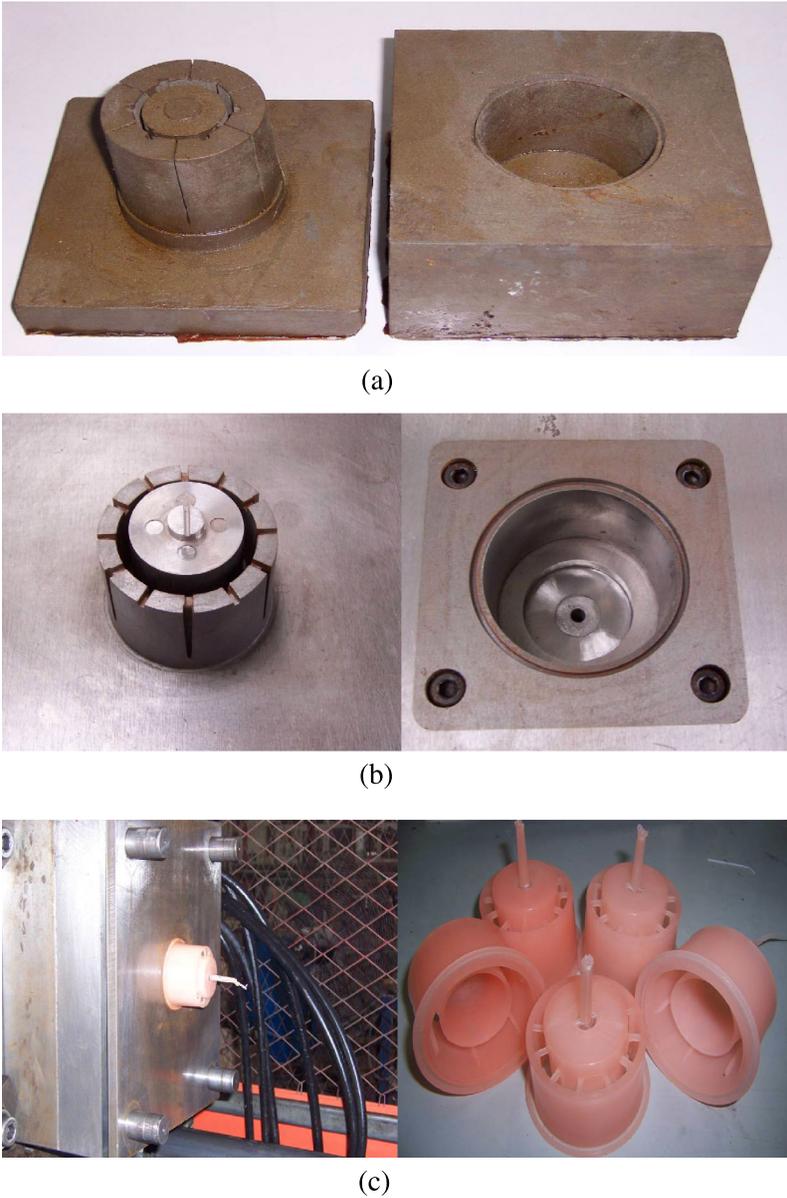


Figure 9. (a) Tooling inserts made of Fe/epoxy resin composites; (b) tooling inserts made of Fe/epoxy resin composites after machining; (c) plastic parts from the injection of the tooling inserts.

(0.050–0.250 mm) of the part, the UV laser beam draws the next slice on a fresh layer of liquid polymer. This slice of the part solidifies on top of the previous set slice. When all the slices have been traced by the UV laser beam, the platform is removed from the vat and excess liquid polymer is cleaned off the completed part. The completed part is then finally cured in an ultraviolet oven [39–41].

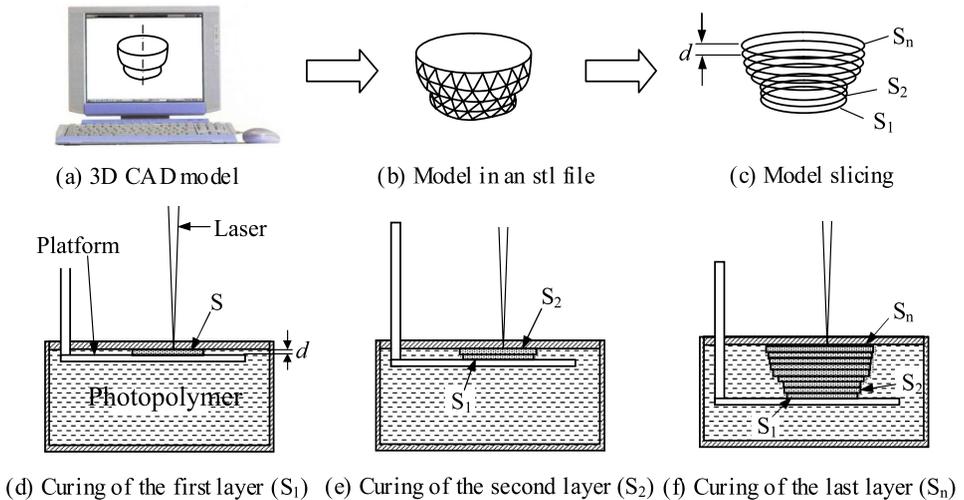


Figure 10. Schematic illustration of the SLA process: (a) 3-D CAD model; (b) model in an stl file; (c) model slicing; (d) curing of the first layer (S_1); (e) curing of the second layer (S_2); (f) curing of the last layer (S_n).

New liquid free radical–cationic hybrid photopolymer, consisting of acrylate-based photo-curable resin and epoxy-based photosensitive resin for SLA by UV laser was developed [42–44]. An acrylate-based photosensitive system (I) consisting of modified epoxy acrylate (EA, 70 wt%), reactive diluent monomer ethoxylated tri-methylpropane (EO-TMPTA, 25 wt%), and irgacure 184 radical photoinitiator (5.0 wt%) was formulated. As a type of cationic resin, the epoxy-based cationic photopolymer (II) was prepared by mixing desired weight quantities of 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate (SarCat K126), 1,4-cyclohexane dimethanol divinyl ether (CHVE), epoxy resin E-44, and a cationic photoinitiator. The hybrid photopolymer system (III) was composed of I (30 wt%) and II (70 wt%). All of these mixtures were vigorously stirred and stored in the dark.

The experimental results indicated that the hybrid photopolymer exhibits the advantages of both the acrylate-based photosensitive resin and the epoxy-based photosensitive polymer contained in the hybrid system with relatively high photospeed and low linear shrinkage. SLA parts without obvious distortion were built on the SLA machines HRPL series from this hybrid resin, successfully and efficiently. Figure 11 shows the SLA parts made from the free radical–cationic hybrid photopolymer with complicated structures.

5. Future Trends

It is clear from the examples described above that there is still much that can be done to incorporate new multiphase polymeric materials into RP and RT technologies and open up new application areas.



Figure 11. SLA parts made from the free radical-cationic hybrid photopolymer with complicated structures.

- (1) Fabricating polymer composite scaffolds for tissue engineering by using RP technologies has become a hot research topic. It is believed that polymer composites have the following advantages for medical applications.
 - Polymers are mostly more compliant with biological hard tissue than metal and ceramics, and can have strengths of the same order of magnitude with hard tissue. It is of great interest in the compounding of polymers and other materials to obtain composites that attain combinations of mechanical and biological properties similar to those of biological hard tissue.
 - Higher variability of designing a targeted degradability and resorbability as well as improved biocompatibility.
 - They can be processed into various three-dimensional shapes and microstructures by using RP and RT technologies.
- (2) The aerospace industry is already focusing much effort into polymer composite parts with complex structures. RP and RT technologies exactly have the ability to construct polymer composite parts with complex geometries.
- (3) Rapid manufacturing (RM) is currently a major goal of many RP vendors and researchers. A problem is that the parts produced by RP are generally inferior in performance compared with similar parts made using conventional manufacturing technologies. RM benefits are mainly in terms of speed, costs and complex structures. It is interest in developing multiphase polymeric materials to enhance parts properties produced by RP and RT technologies.

6. Conclusions

The HIPS/epoxy blend, HIPS/wax blend, PS/PA12 alloy, PA12/OREC and nanosilica/PA12 nanocomposites for the SLS process have been successfully developed, and their SLS components have been made for various applications.

As an RT application, Fe/epoxy resin composite tooling inserts have been successfully fabricated by SLS and post-processing. Green shape of tooling inserts with internal conformal cooling channels is formed by the SLS process from a nylon-12 coated Fe powder. After the post-processing process of binder decomposition, vacuum sintering, infiltration of epoxy, curing and being simply machined, the tooling inserts made of Fe/epoxy composites can be used for plastic injection. Some good quality plastic parts are finally obtained through plastic injection using the mold.

A new liquid free radical–cationic hybrid photopolymer, consisting of acrylate-based photo-curable resin and epoxy-based photosensitive resin for SLA by UV laser was also developed. The SLA parts with complicated structures are made from the free radical–cationic hybrid photopolymer by the SLA machines HRPLA series.

Acknowledgements

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