Materials and Coatings Fabricated Using the Additive Manufacturing Technologies
Материалы и покрытия, получаемые методами аддитивных технологий

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Review article
Обзорная статья

Additive manufacturing of polymer-ceramic materials using fused deposition modeling (FDM) technology: A review

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Abstract. Additive manufacturing technologies, also known as 3D printing, are currently undergoing rapid development and gaining wide popularity, complementing and, in some cases, replacing traditional manufacturing methods. Particular attention is being paid to the fabrication of products from metallic, ceramic, polymeric, and composite materials. Among the seven commonly recognized methods of additive manufacturing, material extrusion stands out, which includes the Fused Deposition Modeling (FDM) technology. The heightened interest in FDM is due to the accessibility of equipment and the wide range of starting materials available, ranging from classic polymers such as PLA and PETG to composite materials, including metal- and ceramic-filled filaments. The objective of this study was to systematize and summarize the existing knowledge on the fabrication process of polymer-ceramic products using ceramic-filled filaments. The paper provides an analysis of the main stages of production, including material selection, filament fabrication, and the 3D printing process. Areas of research and potential applications are also examined.

Keywords: additive manufacturing, 3D-printing, FDM technology, polymer-ceramic materials, filled filament

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Аддитивное производство полимер-керамических материалов методом послойного наплавления материала (FDM-технология): Обзор

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Аннотация. Технологии аддитивного производства, также известные как 3D-печать, находятся в фазе активного развития и набирают широкую популярность, заменяя и дополняя при этом традиционные способы производства. Особое внимание уделяется получению изделий из металлических, керамических, полимерных и композиционных материалов. Среди 7 общепринятых методов аддитивного производства отдельно выделяют экструзию материала (*material extrusion* – MEX), которая включает в себя технологию послойного наплавления материала (FDM). Повышенное внимание к ней объясняется доступностью оборудования и возможностью использования широкого спектра исходных материалов (от ставших классическими полимеров PLA, PETG и др. до композиционных материалов, в том числе метало- и керамонаполненных нитей). Цель настоящей работы заключалась в систематизации и обобщении существующих знаний о процессе изготовления полимер-керамических изделий с использованием керамонаполенных филаментов. Представлен анализ основных этапов производства, выбора исходных материалов, получения филамента и процесса 3D-печати. Рассмотрены области исследований и потенциальные сферы применения.

Ключевые слова: аддитивное производство, 3D-печать, FDM-технология, полимер-керамические материалы, наполненный филамент

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Introduction

In recent decades, there has been significant growth in the development of new materials and manufacturing methods, driving the advancement of additive manufacturing (AM) technologies, also known as 3D printing. These are considered innovative fabrication processes capable of partially replacing or optimizing traditional manufacturing methods [1–4]. An advantage of AM is its ability to reduce material waste during production by building products with diverse geometries layer by layer. This approach allows for creating complex shapes in a single technological process. AM technologies enable scientists and engineers to drive unique innovations through the use of advanced materials and cutting-edge solutions [5–9]. One of these innovations is the implementation of AM processes for producing polymer-ceramic materials [10]. Interest in these materials is driven by the combined benefits of polymers and ceramics: the manufacturing flexibility of polymers and the unique properties of ceramics, such as high strength, hardness, and electrical characteristics.

This review provides a detailed examination of the process for fabricating polymer-ceramic composites (PCC) using FDM (Fused Deposition Modeling) technology (see Fig. 1). An overview of the 3D printing method for polymer-ceramic materials is presented, along with an analysis of the process for obtaining ceramic-filled filament. The specifics of FDM printing with these materials are described, current trends in research and manufacturing of PCCs by 3D printing are highlighted, and conclusions are drawn on the current state of FDM printing with polymer-ceramic materials.

3D printing with polymer-ceramic materials

The production of polymer-ceramic products is advancing, driven by developments in new materials, designs, and components for functional applications. A well-established traditional manufacturing method for these materials is injection molding [11–13]. Among the AM methods used for producing PCC, FDM and stereolithography (SLA) [14–17] are widely applied, along with other technologies [18; 19].

The FDM method has been in use since the late 20^{th} century when the U.S. company Stratasys patented it as "Fused Deposition Modeling" [20; 21]. This method works by sequentially depositing layers of filament, heated to a viscoelastic state, through a nozzle to build up the product layer by layer [22]. Today, many commercially available machines are based on this technology, commonly as desktop systems that

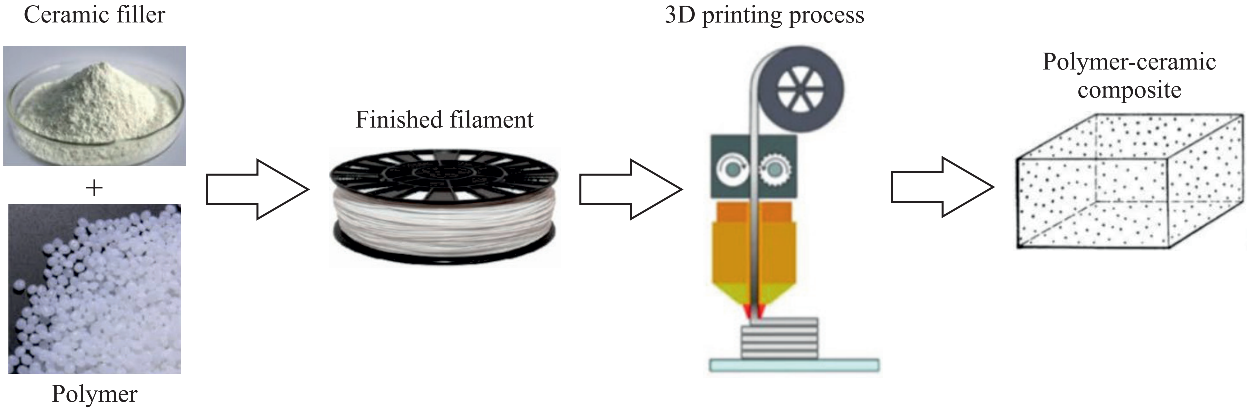


Fig. 1. Key stages in ceramic-filled filament production, from raw material selection to final product

Рис. 1. Основные стадии производства керамонаполненного филамента: от выбора сырья до конечного изделия construct products within the build platform's plane. FDM printers are classified by their material feeding systems, which vary by feeder placement:

– direct extruder, where the feeder is attached directly to the print head;

– bowden extruder, where the feeder is mounted on the printer's frame, and the material is channeled to the print head via a tube [23–25].

Direct extruders are preferred for flexible, brittle, and composite materials as they reduce the risk of nozzle clogging and polymer deformation in the feeding channel during printing.

In FDM technology, polymer filaments of varying diameters, tailored to equipment specifications, are used as feedstock. Currently, many manufacturers offer both standard 3D printing polymers and advanced materials with improved compositions that provide better mechanical strength, wear resistance, shape memory, and higher operating temperatures. Research continues to enhance existing thermoplastic filaments for FDM printing and develop new formulations with improved properties based on various material types [26–28].

Key aspects of producing ceramic-filled filament

Filament production is a crucial step in the fabrication of PCC, as it significantly influences the properties of the final products and affects the entire production cycle – from 3D printing to the finished item.

In the initial stage, the matrix material and functional filler are selected according to the desired characteristics of the final product. The polymer matrix is typically made from common thermoplastic materials used in FDM printing, such as polylactic acid (PLA) [29; 30], acrylonitrile butadiene styrene (ABS) [31; 32], and, less frequently, polyethylene terephthalate glycol (PETG) [33; 34] and polyamide-12 (PA12) [35; 36]. Ceramic fillers often include technical ceramics, such as aluminum oxide (Al_2O_3), silicon dioxide (SiO_2), zirconium oxide (ZrO_2), titanium oxide (TiO_2), and silicon carbide (SiC) [12; 37]. These materials are widely used due to their unique physical, mechanical, electrical, and thermal properties. In addition, piezoceramic powders – such as barium titanate ($BaTiO_3$) and barium strontium titanate ($BaSrTiO_3$) – are used to enhance electrical properties. During material selection, the particle size of the ceramic powder is also determined, as it affects both the properties of the PCC and the quality of 3D printing. Table 1 presents different combinations of ceramic-filled filament materials, including commercially available options and those developed in research studies.

Following the selection of the raw material composition, the next steps are PCC preparation and filament production. The primary stages of this process include creating the composite mixture and manufacturing the ceramic-filled filament for 3D printing. Preparation and intermediate steps often involve moisture removal from the initial materials. Additionally, various additives such as acetone, stearic acid, and others are used to enhance the adhesion of ceramic particles to the polymer and to improve dispersion (see Table 1).

This filament production process is detailed in numerous studies by various research teams. For example, in [40], researchers from the Institute of Nanoscience and Nanotechnology, N.C.S.R. Demokritos (Greece) produced composite filament from PLA granules (Gorinchem, Netherlands) as the matrix, combined with SiC powder (particle size 8.3 μm) (Struers, Denmark). The polymer granules were pre-dried and then mixed with the ceramic powder. Acetone was added to enhance adhesion between the granules and ceramic particles. The prepared mixture was then dried (100 °C for 24 h). The resulting raw material was processed through a single-screw extruder (Felfil Evo, Italy) at 185–195 °C, yielding five types of composite filaments with a diameter of 1.75 mm and varying SiC content (from 1 to 3 wt. %).

A similar combination of materials was chosen by a team of researchers from the Department of Mechanical Engineering at Stevens Institute of Technology, New Jersey, USA [39]. However, instead of polymer granules, PLA (74 μm) and SiC (15 μm) powders were used as the starting materials. Before further processing, the materials were pre-dried at 70 °C for 4 h. Mixing was conducted in a rotary tumbler using chromium steel balls. Filament production was carried out on a single-screw extruder. To further study the dispersion of SiC in PLA, filament samples containing 50 wt. % SiC were analyzed. Based on image analysis (Fig. 2), it was concluded that SiC particles were adequately dispersed within the PLA matrix. This is supported by scanning electron microscopy (SEM) images showing a substantial reduction in ceramic powder particle size from the initial 15 μm to approximately 5 μm during mixing.

A similar filament fabrication method was used in [45], where researchers from the Czech Technical University in Prague (Department of Electrotechnology, Faculty of Electrical Engineering) employed PETG granules and TiO_2 powder (50–300 μm) as starting materials. Filaments with varying TiO_2 content were produced for the study. In two cases, PETG was filled with titanium dioxide (10 and 20 wt. %) to increase

Table 1. **Results of analyzing the process of producing ceramic-filled filament**

Таблица 1. **Результаты анализа процесса получения керамонаполненного филамента**

Polymer	Ceramic	Brief description of filament extrusion technology	Source
PLA	ZrO_2	Material by Zetamix, France	[38]
	SiC	*Starting materials*: PLA powder (74 μm) and SiC powder (15 μm). The powders were dried at 70 °C for 4 h, then mixed in a rotary tumbler at 64 rpm with steel balls for better dispersion. A single-screw extruder was used to produce filament. Melt temperature was 180 °C, and screw speed was 35 rpm. Various filaments were produced with SiC contents of 10, 20, 30, and 40 wt. %. Comparison was made with pure PLA and PLA with added graphite (C) or a SiC + C mix	[39]
		Starting materials: PLA granules and SiC powder (8.3 μm). PLA granules were dried at 100 °C for 24 h, then mixed at 75 °C with added acetone. The resulting mixture was dried at 100 °C for 24 h. Filament was produced using a single-screw extruder. Extrusion temperature was 185–195 °C, and extrusion speed was 50 cm/min. Five composite types were obtained with SiC contents of 1.0, 1.5, 2.0, 2.5, and 3.0 wt. %	[40]
	Si_3N_4	Ceramic-filled filament made of PLA/Si_3N_4 in a mass ratio of 95/5 using a twin-screw extruder	[12]
ABS	$BaSrTiO_3$	*Starting materials:* ABS granules and $BaSrTiO_3$ ceramic powder. ABS granules were dissolved in acetone at a 1:1.5 mass ratio. The mixture was blended with the ceramic powder, poured into molds, and dried at room temperature for 48 h. The resulting composites were granulated and dried at 80 °C for 48 h. Filament production was carried out using an extruder at 220 °C and 60 rpm. The maximum ceramic powder content reached 50.27 wt. %	[41]
	$BaTiO_3$	*Starting materials:* ABS granules and $BaTiO_3$ ceramic powder (3 μm). ABS granules were mixed with $BaTiO_3$ particles in volume ratios of 10, 20, 30, 35, 40, 45, and 50 %, with 1.1 wt. % stearic acid added as a surfactant. The raw material was dried for 24 h at 130 °C. Filament was produced using a Noztek Pro single-screw extruder (UK) at temperatures between 185 and 210 °C	[42]
		Starting materials: ABS granules and $BaTiO_3$ microparticles (<3 μm). ABS granules were dissolved in acetone, then barium titanate was added. The resulting suspension was placed in molds to allow acetone evaporation. Solidified composite sheets were ground and dried at 70 °C. Filament production was carried out using a Noztek Pro single-screw extruder at 190÷210 °C	[43]
		Starting materials: ABS granules and $BaTiO_3$ microparticles. Octyl gallate and dibutyl phthalate were used as surfactants and plasticizers. Filament production was based on the previous work of Castles F. et al. [43].	[44]
PETG	TiO_2	Ceramic-filled filament prepared in collaboration with Prusa Polymers, Czech Republic. *Starting materials*: PETG granules and TiO_2 particles (50–300 μm). PETG granules were mixed with ceramic particles and melted for homogenization. Composite filaments with TiO_2 contents of 10 and 20 wt. % were produced using a screw extruder	[45]
PA12	ZrO_2 Al_2O_3	*Starting materials*: PA12 granules, Al_2O_3, and ZrO_2 powders. A two-step surface modification of the ceramic powders was conducted: etching and chemical treatment. Preliminary drying: PA12 at 50 °C for 10 h, ceramic powders at 150 °C. Compounding was done with a twin-screw extruder, and filament production used a single-screw extruder (Dr. Collin GmbH, Germany)	[46]
PMMA	ZrO_2	*Starting materials:* PMMA and ZrO_2 nanoparticles (20–80 nm). Preliminary drying was conducted at 100 °C for 2 h. Filament production was performed using a Haake Rheomix 252p single-screw extruder (Thermo Fisher Scientific, USA)	[47]

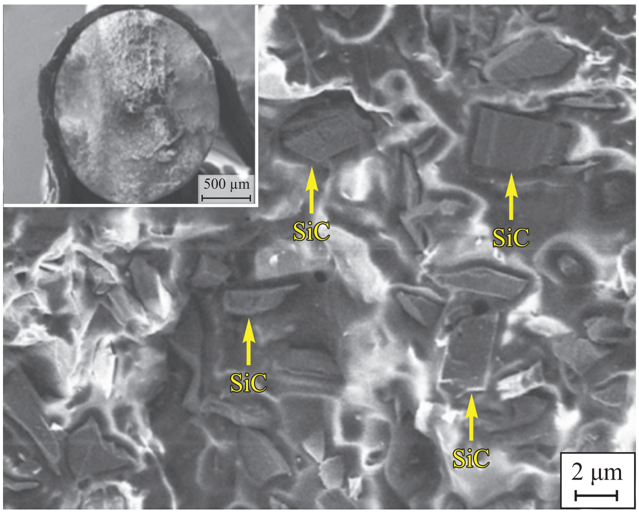


Fig. 2. SEM image of the cross-section of PLA-based ceramic filament containing 50 wt. % SiC [39]

Рис. 2. СЭМ-изображение поперечного сечения керамического филамента на основе PLA с содержанием SiC 50 мас. % [39]

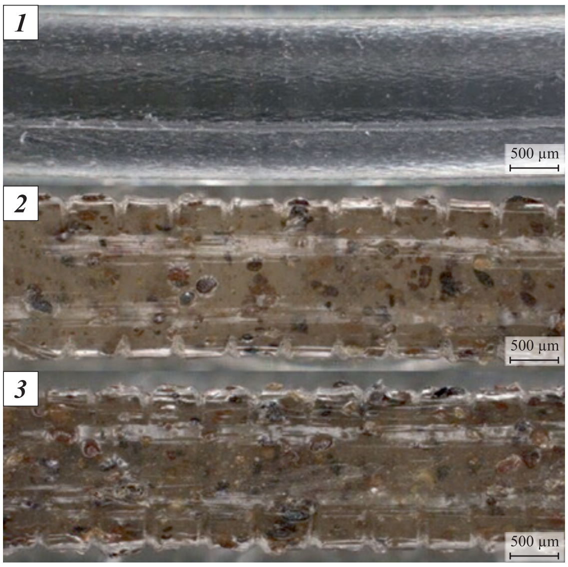


Fig. 3. Microscope images of filaments produced [45]

1 – pure PETG; ***2*** – PETG + 10 wt. % TiO_2; ***3*** – PETG + 20 wt. % TiO_2

Рис. 3. Изображения филаментов, полученные с помощью микроскопа [45]

1 – чистый PETG; ***2*** – PETG + 10 мас. % TiO_2; ***3*** – PETG + 20 мас. % TiO_2

dielectric permittivity, while a third filament was composed of pure PETG (Fig. 3).

The specifics of the process can depend on the methods used to prepare the composite mixture based on the starting components. For instance, study [41] describes a method of producing a polymer-ceramic composite that differs from previous approaches. Researchers in the United Kingdom mixed ABS granules with acetone, dissolving the polymer to create a viscous mixture. To this solution, they added piezoceramic $BaSrTiO_3$ powder (particle size <0.5 µm) and mixed for 10 min. The resulting composite mixtures were poured into specialized molds, where complete solidification occurred over 48 h. The composites were then subjected to mechanical granulation, additional drying, and extrusion to produce filament.

Thus, the analysis presented in this section reflects the essence of producing ceramic-filled filament using various technological methods and materials. This approach is adaptable for working with different combinations of polymer and ceramic compositions, making it one of the most versatile and widely used methods.

3D printing process features

The process of 3D printing with ceramic-filled filaments involves several technological considerations, linked both to equipment specifics and the challenges of working with filled, particularly high-filled, polymers. The particles in the filament increase its brittleness, which can lead to nozzle clogging and filament breakage during printing [48; 49]. Addressing these and other issues in PCC 3D printing is feasible by maintaining optimal conditions, including print temperature, speed, layer height, and feed rate, among others [50–53] (Table 2).

Study [54] explores key FDM printing parameters, including printing speed, track width, layer height, and infill structure (Fig. 4), and examines their effects on the dielectric properties and quality of printed items. The findings indicate that printing speed significantly influences interlayer adhesion and bonding to the build platform. As shown in Fig. 4, *a*, printing speed has a notable impact on surface quality and pore formation, both of which directly affect the final properties of the printed products. A speed range of 10–20 mm/s provided the best print quality, with no visible defects.

Samples were prepared using the specified parameters to measure dielectric characteristics. Results indicated a significant decrease in dielectric permittivity ($\varepsilon_r = 7.38$) compared to a sample produced by injection molding ($\varepsilon_r = 10$). To identify the cause of this discrepancy, surface analysis was conducted using an optical microscope, which revealed air gaps between the print tracks. The solution involved reducing the extrusion width from 0.5 to 0.45 mm (Fig. 4, *c*), which helped increase relative dielectric permittivity and reduce the dielectric loss tangent. Additionally, the influence of layer height on dielectric

Table 2. **Key parameters of FDM printing with ceramic filaments**

Таблица 2. **Основные параметры FDM-печати керамонаполненными филаментами**

Material	Ceramic content, wt. %	Print temperature, °C	Print speed, mm/s	Nozzle diameter, mm	Layer height, mm	Source
PLA/ZrO_2	86	190	40	0.60	0.20	[38]
ABS/$BaSrTiO_3$	50	250	40	0.55	0.10	[41]
PETG/TiO_2	10 and 20	250	–	0.40	0.15	[45]
PLA/Si_3N_4	5, 10 and 15	200	40	0.40	0.15	[12]
PLA/SiC	1–3	200–210	50	1.00	–	[40]
ABS/Ceramic (Premix Oy)	–	260	10–20	0.50	0.30	[54]

characteristics was studied (Fig. 4, *d*), showing that dielectric properties improve as layer height increases. It was also found that adjusting the material infill allows effective control over the dielectric properties of 3D-printed structures (Fig. 4, *e*).

Many studies use commercially available desktop FDM printers. For example, in [45], samples of PETG polymer filled with TiO_2 particles (10 and 20 wt. %) were produced using the I3 MK3S 3D printer by PRUSA Research (Czech Republic), which is equipped with a direct extruder. A 0.4 mm nozzle was used to print samples with a 0.15 mm layer thickness and 100 % infill rate, ensuring high density and minimizing porosity. In study [38], equipment from the same company was selected for fabricating samples from various materials, including PLA with 50 % ZrO_2 particles and polyolefin with different levels of TiO. In this case, a 0.6 mm nozzle was used, with a layer height of 0.2 mm.

An analysis of published studies shows that larger-diameter nozzles are frequently used due to the specific challenges of printing with filled polymers, which can

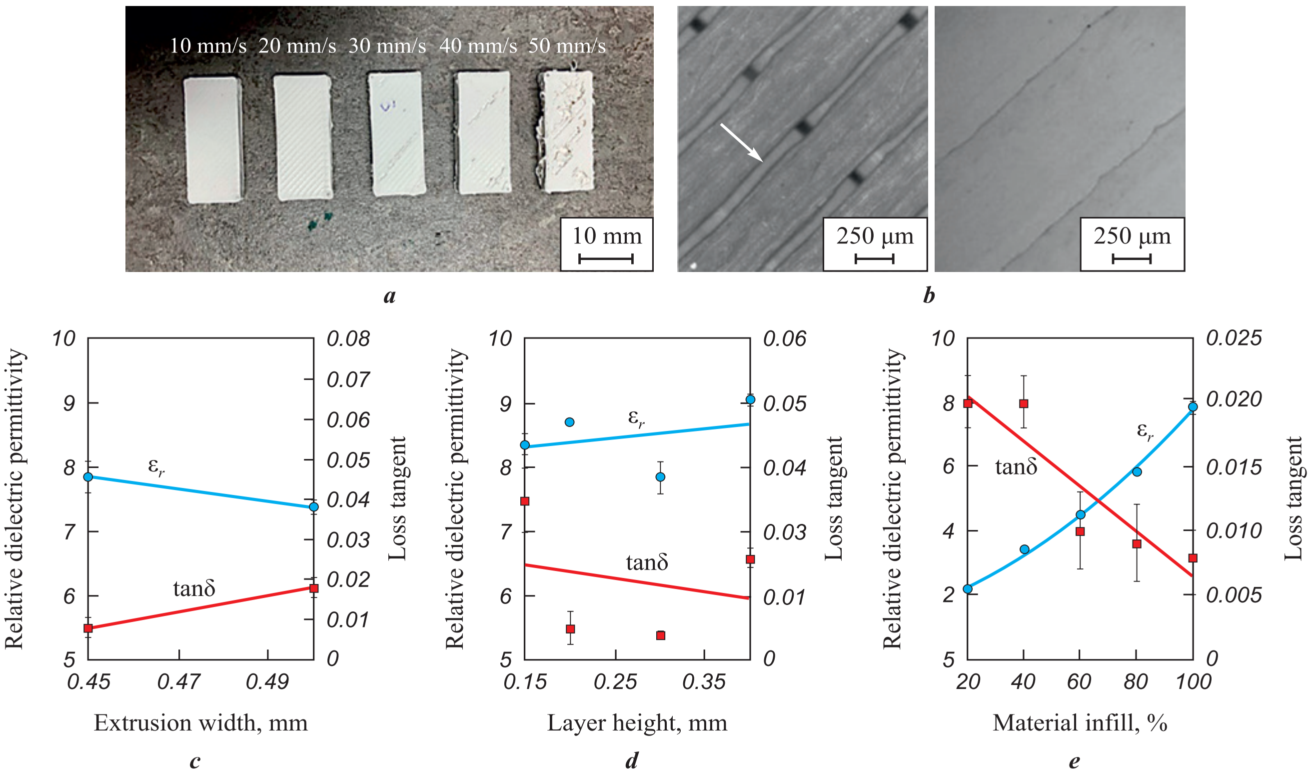


Fig. 4. Research results on the influence of printing parameters on sample quality and dielectric properties [54]

a – influence of printing speed on sample quality; ***b*** and ***c*** – influence of extrusion width on track spacing and dielectric properties; ***d*** and ***e*** – layer height and material infill on dielectric properties

Рис. 4. Результаты исследования влияния параметров печати на качество образцов и диэлектрические свойства [54]

a – влияние скорости печати на качество образцов; ***b*** и ***c*** – влияние ширины экструзии на расстояние между треками и диэлектрические свойства; ***d*** и ***e*** – влияние высоты слоя и коэффициента заполнения на диэлектрические свойства gradually clog the nozzle. Brass nozzles, commonly used in FDM printing, are prone to rapid wear when exposed to ceramic particles, prompting researchers to opt for wear-resistant nozzles. Additionally, nozzle diameter affects the uniformity of material extrusion; increasing the diameter can help reduce the risk of defects (such as pores and cracks) caused by material inconsistencies.

Applications

Research on products made from polymer-ceramic filaments can be grouped into three main areas:

– improving dielectric properties;

– investigating the impact of ceramic fillers on mechanical properties;

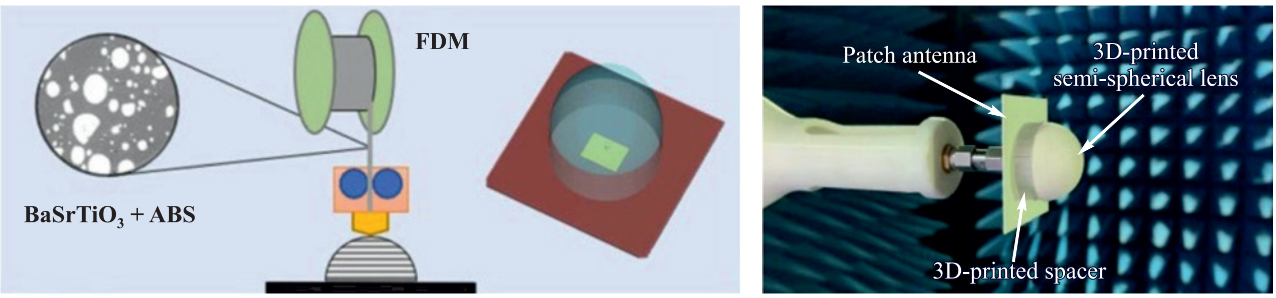
– producing ceramic components from high-filled polymer-ceramic filaments.

A significant portion of research on PCC development using FDM printers focuses on dielectric properties, driven by the ease of fabrication, the broad range of applications, and the growth of 3D-printed electronics. Polymers produced by 3D printing are commonly used as insulators [55]. However, adding conductive carbon fibers or metal particles enables the production of functional parts that conduct electricity [56; 57], while incorporating ceramic powder into the polymer matrix improves dielectric properties. Studies have investigated the effects of TiO_2, ZrO_2, $BaSrTiO_3$ and other ceramic fillers on the dielectric properties of polymer-ceramic samples for application in capacitors, dielectric antennas, and other components that require dielectric materials.

As mentioned in [45], researchers in the Czech Republic investigated PCC made with PETG polymer and TiO_2 ceramic powder. The study involved testing cylindrical samples of different diameters (19.1 and 9.5 mm) and thicknesses (2.8 and 3.0 mm) to assess the dielectric properties of PCC. The sample with 20 wt. % TiO_2 yielded the best results, showing a 50 % increase in dielectric permittivity compared to pure PETG, reaching a maximum value of 4.4. The study found that temperature and frequency had no significant effect on dielectric permittivity or dielectric losses.

In addition to examining the dielectric properties of printed samples, research also explores the potential applications of 3D-printed polymer-ceramic dielectrics in electronic and radio equipment. A team of researchers from the United Kingdom studied PCC properties in [41], using a material based on ABS and $BaSrTiO_3$ powder in a 50/50 weight ratio, which achieved a maximum relative dielectric permittivity ε_r of 6,05. The study also developed a prototype patch antenna (Fig. 5) incorporating a semi-spherical polymer-ceramic dielectric lens. This lens increased the antenna gain by 3.86 dB, with minimal impact on efficiency.

In addition to studies on the dielectric properties of PLA-based polymer-ceramic composites, research is also focused on the impact of ceramic additives on the mechanical strength of the material. In [12], the results of mechanical testing on PLA samples reinforced with Si_3N_4 powder are presented. The researchers compared samples produced by injection molding with those printed on an FDM printer. For the injection-molded samples, three series were prepared with different polymer/ceramic weight ratios (85/15, 90/10, 95/05). Based on mechanical testing results, a 95/05 ratio was chosen for further studies. These samples showed better adhesion and fewer defects, resulting in higher strength values. Using this selected ratio, the mechanical properties of samples produced by both methods were compared. The results indicated that printed samples exhibited reductions in tensile strength, flexural strength, and impact toughness by 12,0, 15.5, and 13.5 %, respectively.



a *b*

Fig. 5. Prototype of the patch antenna

a – process diagram for producing epy polymer-ceramic dielectric lens; ***b*** – appearance of the finished antenna [41]

Рис. 5. Прототип патч-антенны

а – схема получения полимер-керамической диэлектрической линзы; ***b*** – внешний вид готовой антенны [41]

One of the less explored but promising areas in PCC research is the investigation of how ceramic particles affect shape memory effect (SME). It is known that many polymers used in FDM printing can exhibit SME, generally triggered by thermal stimulation [58–61]. Study [39] examined the influence of SiC additives in a PLA matrix on shape recovery characteristics. Results showed that recovery time could be influenced by the thermal conductivity of the material. Tests were conducted on extruded filaments (Fig. 6) and printed samples, revealing that SiC-filled composites recovered shape faster than pure PLA material.

Thus, producing polymer-ceramic composites using FDM technology is an emerging field with several challenges and limitations. Key strategies to address issues such as defect formation (pores, cracks) involve a comprehensive approach that includes achieving an optimal filament composition and selecting appropriate printing parameters.

However, not all issues regarding material homogeneity and defect formation during printing have been resolved. Aspects like the effects of heat treatment and mechanical stresses that occur during printing on product quality and final properties remain insufficiently studied and require further research. These challenges provide a scientific basis for continued study of PCCs and their potential applications in future products.

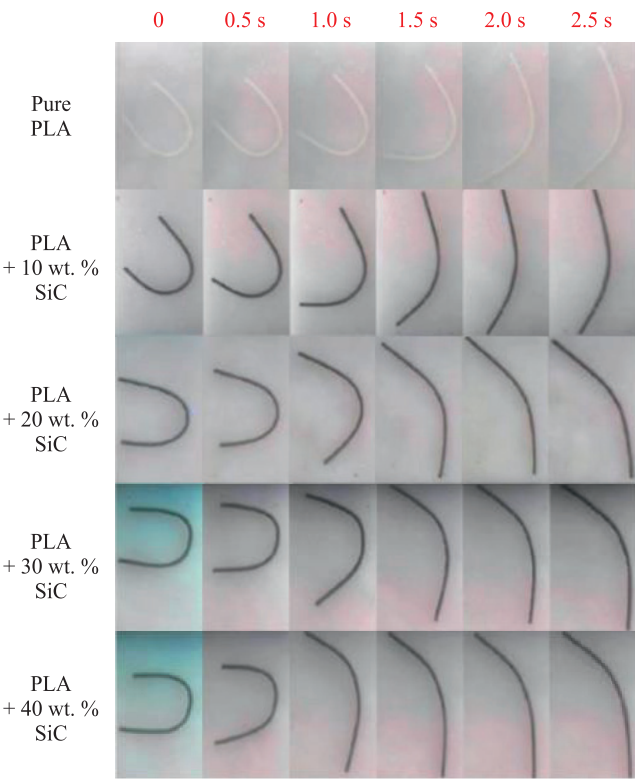


Fig. 6. Images of ceramic-filled filaments showing shape recovery [39]

Рис. 6. Изображения, полученные для керамонаполненных филаментов, показывающие восстановление формы [39]

Conclusion

A detailed analysis of the production of polymer-ceramic composites using FDM printing has been presented, covering key technological stages – from raw material selection to final product creation. A review of scientific publications highlights the most commonly used ceramic additives, such as SiC, ZrO_2, $BaTiO_3$ and others. The use of these fillers improves dielectric permittivity, mechanical strength, and affects the activation time of the shape memory effect. This makes ceramic filaments suitable for creating dielectric components in electronic and radio-frequency systems, sensors, structural elements, and shape-memory products.

The review provides a foundation for further research in the development and study of 3D-printed PCCs. Future work will focus on producing 3D-printed volumetric PCC items with enhanced dielectric permittivity.

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