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Adhesive Bonding of Elements Made Using the Multi Jet Fusion Additive Technique

Abstract: The research discussed the application potential of adhesive bonding in the joining of 3D printed structures made using the Multi Jet Fusion (MJF) state-of-the-art additive technology (HP). The research involved the performance of technological tests aimed to assess the adhesive properties of 3D printout surfaces in the function of a surface layer treatment method as well as to evaluate adhesives used in the joining of 3D printed structures. The tests performed within the research included roughness measurements, contact (wetting) angle measurements, peel strength tests, shear strength tests and bend tests. The results obtained in the tests made it possible to assess the joinability of MJF printouts as well as to identify reasons for problems accompanying the joining of such elements.

Keywords: Multi Jet Fusion, PA12, strength properties, contact (wetting) angle, surface roughness

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Introduction

Recent years have seen the growing popularity of additive technologies and their evolution from the laboratory phase to the phase of production. Many methods, among other things, fused material deposition (FDM) and selective laser sintering (SLS), are increasingly popular in the market of low-volume production, particularly in relation to elements having complex shapes. Additive technologies may revolutionise today's production market, presently dominated by traditional manufacturing techniques such as casting, plastic working or removal machining. Three-dimensional (3D) printing is strictly related to the principal assumption of the industry 4.0 idea, based on individualised customer requirements throughout the life cycle of products and services, ranging from the phase of an idea of a new product or service through the phase of the development and production/implementation to the phase of delivery/rendering and recycling [1].

The making of elements in additive technologies consists in the deposition (adding) of a building material, layer after layer, followed the obtainment of their permanent joint so that a solid element is formed. A model is created in special graphic-design software programmes

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or in conventional 3D software programmes (which, however, requires the conversion of files into those compatible with a printer). It is believed that 3D printing constitutes one of the forms of the transformation of a pixel image into a solid physical object [2].

One of the developmental directions of additive technologies involves the continuation of new manufacturing methods. In 2014, one of the world's largest concerns, i.e. Hewlett-Packard, presented the concept of additive manufacturing referred to as Multi Jet Fusion (MJF) (Fig. 1) [3], where the building material is powder. The presently available commercial offer of materials used in the above-named technology includes, among other things, polyamide PA11 and polyamide PA12. MJF is based on the consolidation of material by means of two additions, i.e. a joining agent and a finishing agent, as well as elevated temperature generated by a system of lamps [3]. The chamber in which a given element is printed is filled with appropriate powder arranged by a roller in the working area and subjected to preheating. Highly precise heads dose drops of the melting and decorating agent (30 million drops per second per each inch of the working area) [3]. Afterwards, the material is subjected to heating again followed by the resultant joining of powder particles. Such a manner of manufacturing provides the user with the full control over the geometry and subsequent properties of elements at the level of single voxels [3]. After the completion of the entire process, the working chamber containing printed solid element requires cooling, taking place in the HP Jet Fusion 3D 4200 processing station, featuring the fast cooling function. The elements are removed from the chamber after reaching appropriate temperature. Other MJF advantages include the significant reduction of waste as the same material can be used several times. In addition, it is not necessary to apply supporting structures during the printing process. The technology patented by Hewlett-Packard is currently the most efficient production method among commonly available additive technologies and has one of the largest working chambers (380 mm × 284 mm × 380 mm) among currently commercially present 3D printing systems [3].

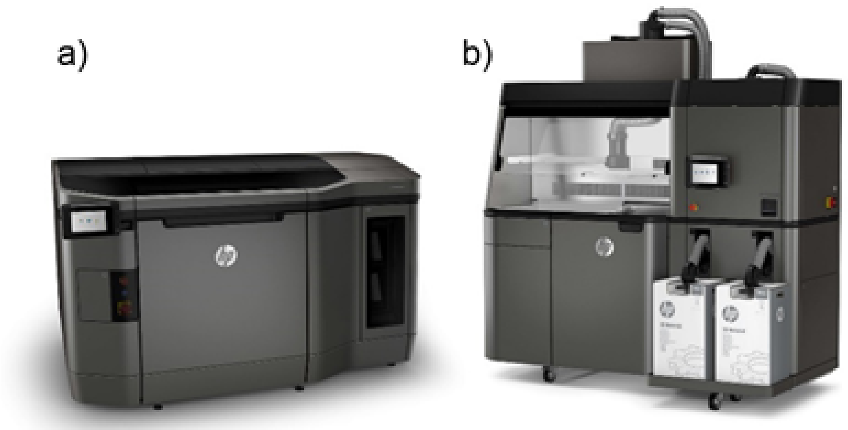

Fig. 1 Printer (a) and the HP Jet Fusion 3D 4200 processing station with the fast cooling function (b) [3]

It is generally agreed that the adhesive bonding of elements in additive technologies poses an additional activity connected with time and expenses. All additive technologies are limited by the size of a working area/surface and, consequently, the dimensions of printed elements. However, the primary objective of additive technologies is the making of a complete and fully functional product in accordance with customer's requirements. Therefore, it is sometimes necessary to join several separate printouts in order to create a complete product. The above-named operations are particularly useful in quick prototyping [4, 5]. Another issue connected with joining is the repair of 3D printouts damaged in excessive operation or of printouts which, because of their complex shape, lost geometric parameters directly during the printing operation. The above-named issues inspired a discussion concerning the possibility of joining individual elements so that they make up a functional finished product. This study involves the analysis and discussion related to the adhesive bonding of 3D printouts made of plastics (polyamide PA12) using additive technologies (Multi Jet Fusion).

Scientific reference publications contain information about adhesive bonding with respect to 3D printed structures, yet this issue is far from being very popular. In terms of 3D printing, adhesive bonding is developed in several directions including the aforesaid joining of prints characterised by larger sizes [4, 5], the design of joints in relation to 3D printed structures [6], printing involving the use of adhesive as one of the building/aiding agents [7], including biomaterials [8] as well as the combination of adhesive bonding and other advanced technologies, e.g. Cutting-Bonder Frame [9, 10].

Materials and methods

All of the test specimens were made using the Multi Jet Fusion technology and an MJF 4200HP device. The test elements were printed in one working cycle using the so-called balanced print setting (the device enables printing in several operating modes), i.e. the most frequently applied programme, ensuring the obtainment of a balanced proportion of surface quality to the mechanical properties of the structure. Afterwards, the surface of the elements was prepared for the adhesive bonding process using abrasive blasting (with glass spheres, the size of which was restricted within the range of 70 μm to 110 μm or electrocorundum F40, having the average size of particles amounting to 425 μm) and abrasive paper having an abrasive grain granularity of P180 (characteristic dimension of cut grains restricted within the range of 90 μm to 63 μm). Because of the fact that abrasive blasting with glass spheres is the dominant method when finishing 3D MJF printouts, it was treated as superior and, depending on tests, compared with other selected technologies.

The roughness of the surfaces to be joined was measured in accordance with the PN-EN ISO 4288:2011 standard using an MarSurf PS 10 profile measurement system. The contact device mapped irregularities of the surface, characterised on the basis of the following parameters: Ra – arithmetic mean of the profile deviation from the mean line measured along the measurement section, Rz – height above the mean line along the measurement section and Rt – total height of the profile.

The specimens used in the tests of adhesive-bonded joints were designed in the CAD programme and based on the guidelines of related standards developed for individual tests. The printouts were made of commercially available polyamide PA12 powder offered by Hewlett-Packard. According to information provided by the manufacturer, the above-named powder is characterised by the globular shape, an average particle size of 60 μm and a bulk density of 0.43 g/cm^3 [4].

The joining process involved the use of adhesives intended for 3D printing, i.e. Loctite 3D Universal Bonder – a two-component hybrid adhesive and Loctite 3D Instant Bonder – a quick-setting one-component cyanoacrylate adhesive, activated with Loctite SF7452 Activator, i.e. an accelerator quickening the setting of cyanoacrylate adhesives. For comparative purposes, selected tests involved the use of two high-strength universal (general purpose) adhesives (also for polymers), i.e. a Distal Classic two-component epoxy adhesive and an Agomet F330 two-component methacrylate resin-based adhesive.

The tests concerning the wettability of surfaces to be subjected to adhesive bonding were performed using an HTM Reetz LOBA/I 1200-53-350-1, i.e. equipment for measuring the angle of the so-called "sitting drop". The above-named equipment was composed of a manually adjustable table for the basing of specimens, an optical system recording and imaging the shape of a drop, a special syringe for dosing the measuring liquid and an adjustable source of light illuminating drops. In the above-presented system for measurements of drop shapes, several drops of liquid are applied on a specimen using a needle. The shape of a drop is recorded by a digital camera and, afterwards, by means of a dedicated software programme, is magnified and subjected to analysis. The analysis can be based on several methods including globular matching, height and width measurements as well as the tangent method. The result of the test is the value of specimen contact angle (contact angle) in relation to given liquid. The tests involved 2 flat specimens having dimensions 40 mm × 40 mm × 3 mm. The elements were subjected to two types of the abrasive blasting treatment, i.e. one involving the use of glass spheres, whereas the second one performed using electrocorundum. In the experiment, the contact angle was tested by means of water and diiodomethane. Drops of liquid (of specific volume) were applied on the model surface using the needle. To ensure the repeatability of results, the test was repeated 5 times. Afterwards, the optical system and the dedicated software programme were applied to calculate the contact angle using the globular matching method.

The peeling and shear tests of the adhesive-bonded joints were performed in accordance PN EN ISO 178 and PN EN ISO 14869-2: 2011, using two devices, i.e. Elcometer 510 and an M100-1CT testing machine respectively. The shape and dimensions of the elements subjected to the strength tests are presented in Figures 2 and 3.

The bend tests involved the specimens having dimensions presented in Figure 4. The three point method-based bend tests were performed in accordance with PN-EN ISO 178. The mandrel and supports (located 64 mm away from one another) had a diameter of 10 mm. The beam travel rate amounted to 2 mm/min.

Test results

The roughness measurement results (being the arithmetic mean of 3 independent measurements) are presented in Table 1.

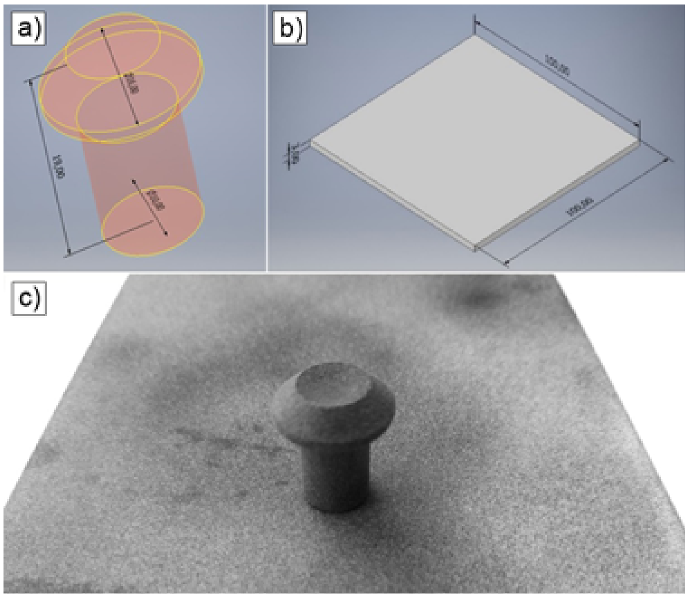

Fig. 2 Model (a, b) and the actual shape (c) of the specimens used in the peeling test

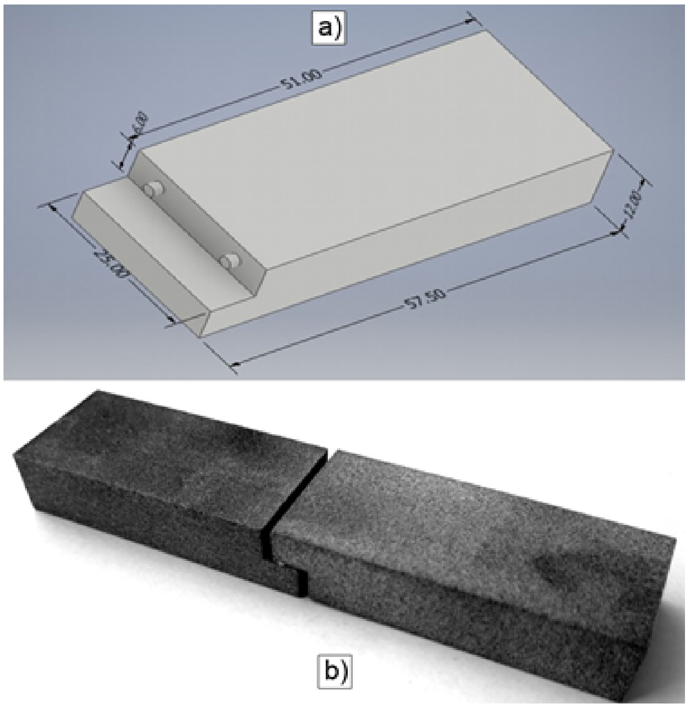

Fig. 3 Model (a) and the actual shape (b) the specimens used in the shear test

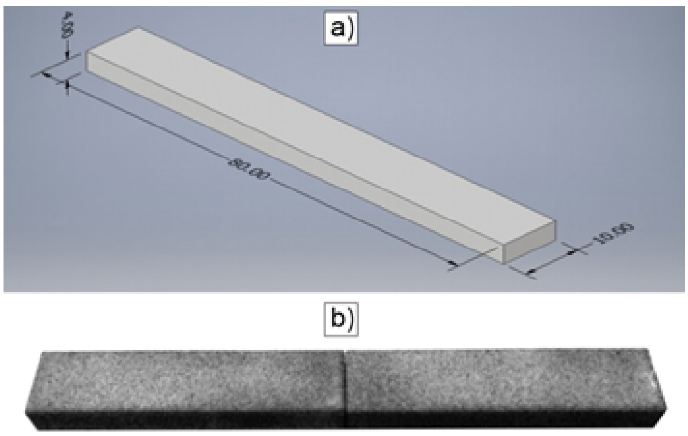

Fig. 4 Model (a) and the actual shape (b) the specimens used in the bend test

Table 1 Average results of roughness measurements related to working surfaces of the specimens

Finishing treatment / Roughness parameters	Finishing abrasive blasting with glass spheres,	Finishing abrasive blasting with electrocorundum	Finishing with abrasive paper P180	Without finishing treatment
Ra	9.5±1.4	8.6±0.6	5.5±0.4	12.4±1.0
Rz	50.9±7.8	48.6±1.6	37.7±3.1	69.9±5.3
Rt	65.5±11.7	69.0±6.16	47.0±6.0	90.1±5.7

According to data contained in related reference publications, the most favourable roughness of a surface to be subjected to adhesive bonding, expressed by parameter Ra, should be restricted within the range of 1.6 μm to 6 μm. In turn, value Rz should be restricted within the range of 15 μm to 40 μm [11–13]. Nearly all of the results obtained in the tests exceeded the above-presented values, which, in terms of adhesive bonding, could create problems. The excessively high roughness of the surface favours the formation of a notch and limited wetting by high viscosity adhesives, leading to the confinement of air in cavities [14–16]. The foregoing combined with the low surface energy of polyamide (36 mJ/m2) [17] leads to the formation of air pockets between surface irregularities and the adhesive, favours the non-uniform spreading of the adhesive and, consequently, reduces the mechanical properties of joints. Only the results of the surface treatment involving the use of abrasive paper were restricted within recommended ranges, yet very close to limit values.

The results of contact angle measurements (averaged on the basis of 3 independent measurements) are presented in Table 2. Differences between water and diiodomethane-related indications resulted from their properties and interaction with polymer. The measured contact angle values revealed that more effective was the treatment with electrocorundum, providing deeper penetration into the base material and removing the poorly wettable upper layer (composed primarily of unmelted powder PA12) from the surface. However, it was also possible to observe that, regardless of the surface preparation method, the wettability of polymer PA12 substrate was ultimate. In relation to adhesive bonding, the exceeding of 30° entailed potential wettability-related problems and, as a result, the possible non-existence of adhesive interaction [11]. The tests revealed another problem, i.e. variable directivity depending on a given substrate area.

Table 2 Results of contact angle measurements

Wetting liquid	Finishing abrasive blasting with glass spheres	Finishing abrasive blasting with electrocorundum
diiodomethane	33°	22°
water	38°	33°

The peeling tests of pins were performed in relation to three appropriately selected adhesives and verified using two additional high-strength structural adhesives. The most favourable results, regardless of a surface preparation method, were obtained for a Loctite 3D Instant Bonder adhesive with a Loctite SF 7452 activator. The results obtained in the tests were restricted within the range of 2 MPa to 4 MPa, which is not a high value even in relation to adhesive bonding. Therefore, in order to eliminate a possible reason for the poor performance resulting from improperly selected adhesive, the above-named results were compared with those obtained in relation to proven high-strength adhesives (Table 3). The comparative tests produced similar results, therefore the reason for the low strength (in accordance with the previous contact angle results) was found in the weak adhesive interaction at the adhesive-printed

Table 3. Averaged results of tests involving the peeling of the pins off the substrate prepared using various treatment methods

Finishing method	Instant Bonder	Universal Bonder	Instant Bonder +7452	Distal Classic	Agomet F330
	Peel strength [MPa]				
Glass spheres	3.1±0.2	2.1±0.3	3.7±0.2	3.5±0.2	2.9±0.1
Electrocorundum	3.0±0.1	1.6±0.1	3.8±0.5	-	-
Paper P180	2.2±0.2	1.5±0.1	3.3±0.4	-	-

substrate interface, resulting from the poor wettability of the plastic surface, limiting the formation of physical bonds in the boundary zone. The above-named issue is typical of the adhesive bonding of polymer elements made using additive technologies. Depending on a printing technology, the surface layer is usually characterised by less favourable adhesive properties in relation to the core, particularly as regards additive powder technologies (including MJF), where the boundary layer being in direct contact with loose powder may not be properly melted. The most objective test concerned with the usability of adhesives is the shear test of overlap joints (i.e. the recommended form of adhesive-bonded joint design). Table 4 presents average (5 specimens for each adhesive) results of strength tests in relation to the specimens subjected to treatment with glass spheres (i.e. the most popular treatment method involving 3D printouts in the MJF technology). In spite of imperfections in the quality of the surface to be subjected to adhesive bonding (roughness, wettability), the activator-aided Instant Bonder adhesive exceeded a value of 20 MPa, which in the case under analysis should be regarded as a satisfactory result. In each case, the failure was of adhesive nature, i.e. the adhesive got detached from one of the joined surfaces.

Table 4 Results of the shear tests of the adhesive-bonded joints

Adhesive	**Shear strength [MPa]**
Universal Bonder	11±1.0
Instant Bonder	16±1.3
Instant Bonder + activator SF 7452	21±1.2

The verification of the behaviour of the adhesive-bonded joints under conditions of improperly designed shape of elements being joined necessitated the performance of a critical three-point bend test. Because of the significant deformations of the polymer specimens, the adopted test criterion was the value of the joint failure angle. The measured values of angles are presented in Table 5 (average based on 3 tests). The specimens underwent failure at small angles (max. 15°). Slightly more favourable values were obtained in relation to the specimens subjected to the treatment with glass spheres. The test demonstrated the superiority of Instant Bonder, yet without the explicit effect of the activator addition. In each case, the failure was of adhesive nature, i.e. the adhesive got detached from one of the joined surfaces.

Table 5 Results of the shear tests of the adhesive-bonded joints

Treatment / Adhesive	Bend angle	
	Abrasive blasting, glass spheres	Abrasive blasting, electrocorundum
Universal Bonder	8°	6°
Instant Bonder	15°	10°
Instant Bonder + activator SF 7452	15°	10°

Concluding remarks

The analysis of reference publications and results of individual tests justified the formulation of the following concluding remarks:

– The results concerning the roughness of surfaces measured using parameters Ra, Rt and Rz mostly exceeded values recommended in reference publications, which could create problems with the generation of adhesive interactions in adhesive bonding practice. Treatment parameters should be adjusted so that to ensure the optimum wettability in relation to a properly expanded surface.

- The wettability of structures printed in the MJF technology and subsequently subjected to abrasive blasting with glass spheres is at most satisfactory (values above 30°), yet it can also be unsatisfactory. In all of the tests, the rupture was of adhesive nature, which confirmed the thesis concerning the poor wettability of the surface by the adhesives. The authors recommend the performance of a series of additional tests involving more efficient (in terms of energy) surface treatment (chemical/plasma).
- The peel strength of the printed specimens was restricted within the range of 2 MPa to 4 MPa, which should be regarded as a low value, confirming previous surface analysis (roughness, wettability).
- In the shear tests, the activator-aided Instant Bonder adhesive reached a value >20 MPa, which in the case under analysis should be regarded as a satisfactory result. The foregoing confirms that a properly designed joint, in spite of unfavourable energy-related surface properties, was capable of transferring significant loads.
- The bend tests confirmed that the joints lacked resistance to the effect of unfavourable peeling/splitting forces. The maximum value of the critical angle, the exceeding of which resulted in rupture amounted to 15°.
- Because of the fact that the adhesive bonding of printed structures, particularly as regards powder methods, is problematic, producers of adhesives, in respect of developmental prognoses of 3D printing, should intensify their works on adhesives used in the aforesaid technology. The aforesaid conclusion is confirmed by the results of the tests, where the most favourable strength-related parameters were obtained in relation to the activator-aided Instant Bonder adhesive.

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