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Surface modification as a technique to improve inter-layer bonding strength in 3D printed cementitious materials

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Abstract

The structural capacity of 3D printed components mainly depends on the inter-layer bonding strength between the different layers. This bond strength is affected by many parameters (e.g. moisture content of the substrate, time gap, surface roughness,..) and any mismatch in properties of the cementitious material may lead to early failure. A common technique to improve inter-layer bonding strength between a substrate and a newly added layer is modifying the substrate surface. For the purpose of this research, a custom-made 3D printing apparatus is used to simulate the printing process and layered specimens with a different delay time (0 and 30 minutes) are manufactured with different surface modification techniques (wire brushing, addition of sand or cement and moisturizing substrate layer). The surface roughness was measured and the effect of the modification technique on the inter-layer-bonding strength was investigated. Results showed that the most effective way to increase the inter-layer bonding is increasing the surface roughness by a comb. This creates a kind of interlock system that will provide a higher inter-layer strength. The compressive strength is most influenced by the addition of cement, where the changing W/C-ratio will create a higher degree of hydration and consequently a higher strength.

Keywords: 3D printing; Surface modification; Inter-layer bonding

1 Introduction

The extrusion-based 3D printing technique is a new technology under development for construction of buildings and complex geometries without the use of expensive formwork. When applying this layer-by-layer fabrication method, the layers must bond firmly, to ensure a homogenous structure, as there is no vibration or external force during deposition [1]. Previous research [2-4], focussing on the bonding between fresh and hardened concrete interfaces, namely repair mortars applied on pre-treated hardened concrete surfaces, showed that mainly because of stress concentrations at the interface and the creation of voids between two subsequent layers, the chance of failure at the interfaces is higher. Consequently, these interfaces are able to form a weak link in the overall construction and can affect the structural stability in a negative way. In case of concrete printing, where the situation is slightly different as it concerns a fresh-fresh concrete interlayer, the quantity of influencing parameters increases. Depending on the printing procedure used, the inter-layer bonding strength will also be affected by factors related to the print process parameters, surface roughness and quality, moisture content of the fresh deposited layers, inter-layer time gap and the concrete composition. Consequently, compared to previous research on repair mortars, the occurrence of chemical bonding as well as the effect of restrained shrinkage (caused by different curing conditions) and a changing stiffness over time are phenomena that cannot be neglected in case of 3D printing [5-7].

In general, different techniques can be used to improve the inter-layer bonding strength and within this research, both the effect of an improved chemical and mechanical bonding will be investigated by applying different surface modification techniques. These results will be correlated with the mechanical performance of the cementitious material.

2 Materials and methods

2.1 Materials and mix composition

Ordinary Portland Cement (CEM I 52.5 N) was combined with standardized sand with a maximum particle size equal to 2 mm. To increase the flowability of the cementitious mixture, a polycarboxylic ether (PCE) with a molecular weight of approximately 4000 g/mol and 35% solids was used as a superplasticizer. The mix composition can be found in Table 1.

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Table 1. Mix composition.

Material [-]	Amount [g]
CEM I 52.5 N Strong	675
Sand	1350
Water	246
PCE	0.15% [weight of cement]
ρ [kg/m³]	2200

The mix design aimed to meet the 3D printing requirements, including extrudability, buildability and workability. To classify the composition as extrudable, two demands have to be fulfilled. First, the extrudability was tested by extruding one layer with a length of 300 mm. Once the mortar was expelled without blocking or segregation, the composition met the first requirement. Second, the deformation of the layer after extrusion was considered and deformations within a range of 10% were accepted. The buildability requirement was obtained when at least 5 layers of material could be printed on top of each other. According to previous research [8, 9], this requires a yield stress of the bottom layer $\tau_{0,0}$ [N/mm²] of at least 8920 N/mm² after printing 5 layers. This value was calculated based on the layer height h [mm], density of the cementitious material ρ [kg/m³] and gravity constant g [m/s²] (Eq. (1)).

$$\tau_{0.0} = \frac{\rho g h}{\sqrt{3}} \quad (1)$$

2.2 3D printing process

A custom-made apparatus was used to simulate an extrusion-based 3D printing process (Fig.1). The developed system is capable of printing up to 300 mm long mortar layers, at different speeds and different time gaps between the deposition of two subsequent layers. The nozzle of the print equipment has an elliptical shape (28 mm x 18 mm). The height of each layer is equal to 10 mm with an average layer width of 30 mm. For the purpose of this study, the printing speed is kept equal to 1.7 cm/s.

Sample preparation consists of filling the 3D printing apparatus and extruding material through the nozzle with a constant speed. A single base layer, with a length of approximately 300 mm, was extruded for each specimen. After a predetermined time interval (0 or 30 min), another layer was deposited on top of the previous one. In case of a zero time gap, the two layers were printed form the same batch of material. In case of a 30 min time gap, a fresh mortar mix was deposited on the first layer with a 30 min interval.

To investigate the effect of surface modification, four different techniques were applied. For the first test series, a predefined amount of cement (CEM, 0.16 g/cm²) was manually distributed on top of the first deposited layer. The cement powder will not affect the surface roughness but will act as a bonding agent between the layers. For the second series, a comb (COMB) equipped with 34 small needles (length = 40 mm, Ø = 1 mm) placed next to each other or sand layer (SAND, D_{max} = 2 mm) was used to increase the surface roughness in a non-automated way. These three surface modification techniques were employed directly after deposition of the first layer, without taking into account the predefined time gap. The last modification, in which the first deposited layer was moisturized (WATER), was applied just before printing the second layer.

The before mentioned two layered specimens were used to investigate the mechanical properties of the printed samples, while surface roughness measurements and moisture content measurements were conducted on an individual single base layer. All the specimens were cured afterwards in standardized conditions (20 ± 3°C, RH = 65 ± 5%) until the day of testing (i.e. 28 days after printing).

2.3 Surface roughness

The surface roughness of the printed specimens was determined by an Automated Laser Measurement (ALM) technique, scanning the surface of the layers with a high precision beam, equipped with two stepping motors controlling the motion in X and Y direction. The profile measurements are used to calculate the center-line roughness (R_a) value of the printed specimens. This value is determined with an average line drawn through the profile and the center-line over a selected reference length (200 mm in Y-direction, 15 mm in X-direction). Using ALM, R_a-values with an accuracy of 7 µm can be derived. The surface roughness was measured every 5 and 50 mm in respectively X- and Y-direction (Fig.2). Roughness measurements were performed for every surface modification technique and the results showed in Table 3 are the average values of 5 measurements in X-direction and 4 measurements in Y-directions on different positions.

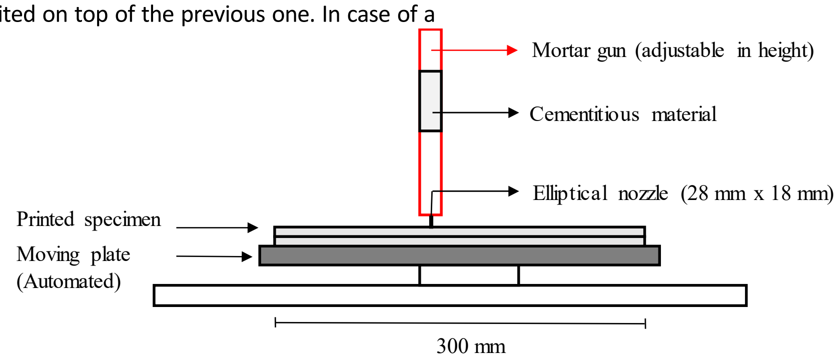

Figure 1. Schematic representation of the 3D print apparatus.

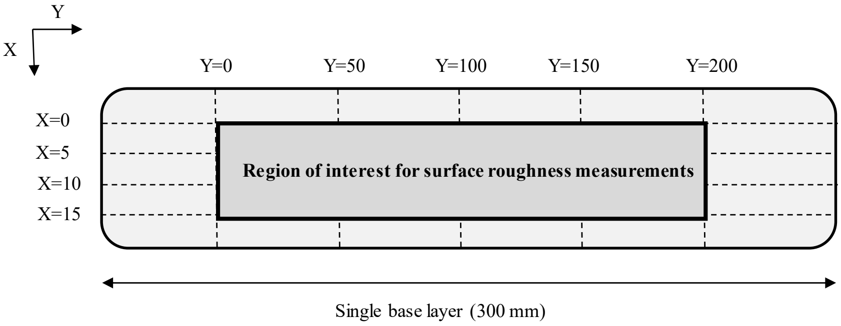

Figure 2. Schematic representation of the surface roughness measurements (ALM).

2.4 Moisture content

The moisture content of every single base layer was measured by covering the layer with a paper towel for 20 sec after the predefined time gap (Eq. (2)). The mass change of the paper was measured as the surface moisture content (Eq. (3)).

$$\text{Moisture content}_{\text{time gap}}[\%]=\frac{M_{20s}}{M_{0s}} \quad (2)$$

$$\Delta_{0\text{-}30}=\text{Moisture content}_{30}\text{-Moisture content}_{0} \quad (3)$$

In case of test series with CEM or SAND, the paper towel was dried afterwards to exclude the weight of cement and sand particles that were attached on the paper. For every delay time or surface modification technique, this test was performed in standardized circumstances (20 ± 3°C, RH = 65 ± 5%). For each time gap or modification technique, three measurements were recorded.

2.5 Mechanical properties

Compressive strength

The compressive strength of the specimens was tested in a universal testing machine under load control at a rate of 100 N/s (Fig.3). For the compressive strength test, small cylinders were drilled from the original printed elements with a height and diameter respectively equal to 20 and 25 mm (Fig.4). Due to the fact that the samples were very small, a hardboard is used during the testing program. This was placed on top and bottom of the sample to deal with the main irregularities. The specimens were loaded perpendicular to the print direction and the anisotropic behavior was not taken into account. At least 3 specimens were tested for every time gap and every surface modification technique.

Figure 3. Compressive strength sample setup.

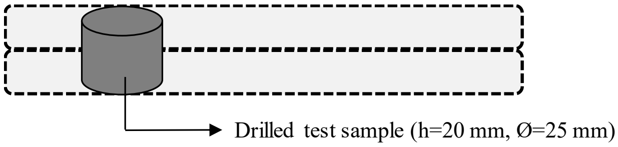

Figure 4. Schematic illustration of sampling element.

Inter-layer bonding strength

Inter-layer bonding was tested on small cylinders, drilled from the original printed elements and with analogue dimensions as the ones used in for measuring the compressive strength (h = 20 mm, Ø = 25 mm). The sample setup (Fig.5) consisted of two metallic brackets which were glued with epoxy on the top and bottom of the printed specimens. The inter-layer bonding strength test was conducted in an universal testing machine under displacement control at the rate of 50 N/s. Special attention was taken to align the specimens in order to avoid any eccentricity. At least 3 specimens were tested for every time gap and surface modification technique. Only test specimens who failed at the interface were taken into account to determine the bonding strength.

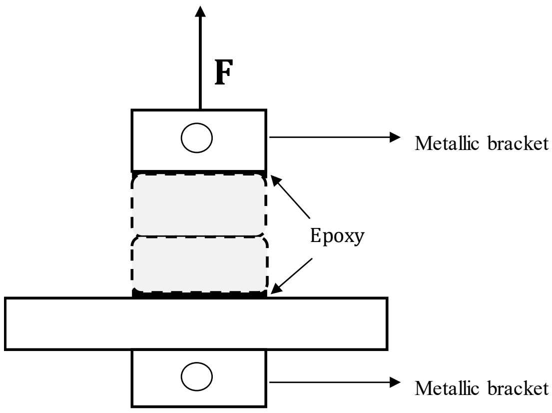

Figure 5. Schematic representation of the inter-layer bond sample setup.

3 Results and discussion

3.1 Moisture content

Table 2 represents the moisture content, measured at different time gaps and for different surface modification techniques. One can see that increasing the surface roughness with a comb or sand layer has almost no influence on the moisture content compared with the reference ($\Delta_{0\text{-}30}$ = 0.08). The addition of cement has a bigger influence and will reduce the moisture content to a higher extent ($\Delta_{0\text{-}30}$ = 0.12). This is due to the fact that the additional cement particles will react with the water of the substrate layer. Increasing the time gap will also increases the reaction degree of the cement particles and consequently, the moisture content of that specific layer decreases. Moisturizing the substrate layer will keep the moisture content equal for both time gaps ($\Delta_{0\text{-}30}$ = 0).

Table2. Moisture content at different time gaps and surface modification techniques.

	T = 0			T = 30			
	M_{0s}	M_{20s}	Moisture $content_0$	M_{0s}	M_{20s}	Moisture $content_{30}$	$\Delta_{0\text{-}30}$
	[g]	[g]	[%]	[g]	[g]	[%]	[-]
REF	0.45	0.51	1.13	0.45	0.48	1.06	0.08
CEM	0.45	0.50	1.12	0.45	0.45	1.00	0.12
COMB	0.45	0.51	1.13	0.45	0.47	1.05	0.08
SAND	0.45	0.50	1.12	0.45	0.47	1.04	0.08
WATER	0.45	0.56	1.25	0.45	0.56	1.25	0.00

3.2 Surface roughness

Table 3 gives an overview of the measured R_a-values of the printed specimens with different surface modifications. The results in X-direction show the average values of 5 measurements, in Y-direction 4 measurements on different positions were performed.

Table 3. Surface roughness measurements.

Surface modification	$R_{a,x}$ [-]	$R_{a,y}$ [-]
REF	0.76	0.20
CEM	0.74	0.20
COMB	0.89	0.29
SAND	1.28	0.33
WATER	0.70	0.18

Based on these results, one can conclude that the addition of sand particles with a maximum particle size of 2 mm affects the surface roughness the most, especially in X-direction. Modification with a comb will also increase the surface roughness, but not in a significant way. The difference between the centre-line and the grooves created by the comb will be smaller than the difference between the centre-line and the sand particles. The addition of water will smoothen the surface. This results in a lower surface roughness, both in X- and Y-direction. The additional cement layer has no influence on the surface roughness.

3.3 Mechanical properties

Compressive strength

Fig. 6 represents the compressive strength and associated standard deviation for the series with different surface modifications and time gaps. One can see that only in case of using a comb, the compressive strength is comparable with the reference sample when the time gap is equal to 0 min. This result is comparable with the ones obtained by [10]. All the other surface modification techniques decrease the compressive strength with approximately 25% (Table 4).

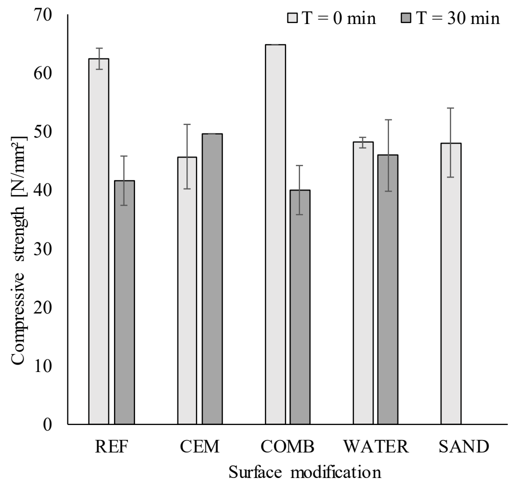

Figure 6. Compressive strength.

Table 4. Compressive strength and strength loss.

	T = 0 min		T = 30 min	
Surface modification	σ [N/mm²]	Loss Δ [%]	σ [N/mm²]	Loss Δ [%]
REF	62.35	-	41.63	-
CEM	45.65	26.78	49.57	-19.07
COMB	64.75	-3.85	40.10	3.68
WATER	48.19	22.71	45.93	-10.33
SAND	48.11	22.85	-	-

Increasing the time gap between the deposition of two layers will generally decrease the compressive strength and the biggest strength loss is observed in case of surface modification with a comb. Only the addition of cement will increase the compressive strength at higher time gaps. This is due to the fact that the additional amount of cement was able to react in a higher extent with the water of the first deposited layer, changing the hydration degree of the layer and consequently increasing the compressive strength. The smallest decrease in compressive strength with an increased layer interval time can be observed in case of a surface modification with water. Based on Table 4, one can generally conclude that after a 30 min time gap, the surface becomes drier due to the evaporation of water. Consequently, this evaporation will lower the amount of water available for cement hydration at the interface resulting in a lower strength. When an extra amount of water is added to the surface, the water amount available for the hydration process increases resulting in a stronger interface zone, a higher amount of capillary pores and consequently also a smaller decrease in the compressive strength (Fig. 6). In order to state this conclusion, more detailed investigations by means of Scanning Electron Microscopy (SEM) are required and will be performed in further research.

The strength loss mentioned in Table 4 is given as the ratio of 'difference in compressive strength with certain modification technique' to the compressive strength of the reference sample without surface modification (Eq. (4)).

$$\Delta = \frac{\text{Strength}_{\text{REF}} - \text{Strength}_{\text{SURFACE MODIFICATION}}}{\text{Strength}_{\text{REF}}} \quad (4)$$

Inter-layer bonding strength

Fig. 7 shows the positive effect of the grooves created by the comb on the inter-layer bonding strength. These grooves will provide an interlock system between the different layers, increasing the inter-layer bonding strength with 21% (Table 5).

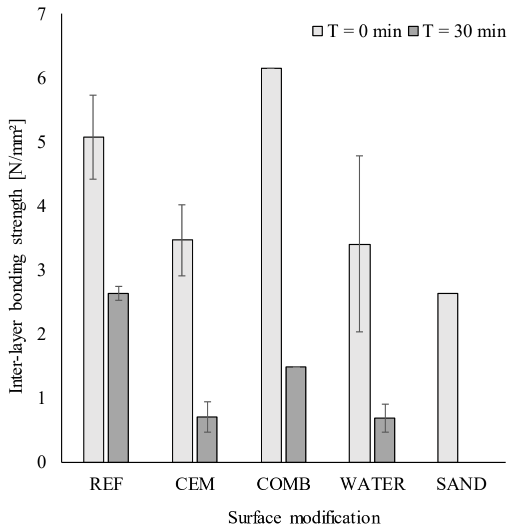

Figure 7. Inter-layer bonding strength.

Although the highest surface roughness is created after a surface modification with sand, this technique decreases the inter-layer bonding strength to the highest extent (48%) in case of a 0 min time gap and even creates a cold joint in between the printed layers when increasing the time gap further. The sand particles, which were added in a non-automated way, did not attach properly to the first deposited layer, creating large cavities in between the first and second layer of the printed specimen. This weakened the interface zone and resulted in a lower inter-layer bonding strength. The creation of these cavities was also visible after sample drilling and is even more pronounced in case of a higher time gap. In case of a 30 min time gap, it was not possible to measure the inter-layer bonding strength of these samples. The energy required to drill the cylindrical samples was so high that the layers detached during the drilling process. Based on Fig. 7, one can also conclude that none of the applied modification techniques improves the inter-layer bonding strength in case of a higher time gap. This conclusion implies that the surface modification techniques used in this research should be further developed and optimized in order to improve the bonding also in case of a higher time interval. The strength loss, mentioned in Table 5, is calculated based on Eq. (4).

Table 5. Inter-layer bonding strength and strength loss.

	T = 0 min		T = 30 min	
Surface modification	σ [N/mm²]	Loss Δ [%]	σ [N/mm²]	Loss Δ [%]
REF	5.08	-	2.63	-
CEM	3.47	31.74	0.71	73.08
COMB	6.16	-21.32	1.48	43.70
WATER	3.41	32.85	0.69	73.72
SAND	2.63	48.19	-	-

4 Conclusions

The effect of different surface modification techniques on the mechanical properties of 3D printed cementitious materials was investigated in this research. The mix composition and printing speed was kept constant during all the experiments. The following conclusions can be drawn from this study.

1. The moisture content of the surface decreases the most in case an additional amount of cement is distributed on the first deposited layer.
2. Surface modifications with a comb or through addition of sand increase the surface roughness of the substrate the most compared to the reference one. Moisturizing the samples creates a smoother surface. The difference in surface roughness is more pronounced in X-direction than in Y-direction.
3. The compressive strength in case of a zero time gap is affected in a negative way (strength loss of approximately 25%) when applying a certain modification technique. Only the samples with grooves, created by a comb, show comparable results with the reference. Increasing the time gap will generally decrease the compressive strength. However, an additional amount of cement results in higher compressive strengths due to the higher reaction degree between the cement particles and the water of the substrate layer.
4. The inter-layer bonding strength is positively influenced through the use of a comb, due to the interlock that is created between the two layers. The higher surface roughness, created by the sand particles, is not enough to ensure a proper bonding between two subsequent layers.
5. Increasing the surface roughness has more impact than moisturizing the substrate.

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