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Improvement of the adhesion in functional NiTi wire/polymer composites made by additive manufacturing

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A B S T R A C T

Additive manufacturing allows the integration of shape memory wires into polymer matrices. Actuators produced in this way offer a wide range of shapes and wire integration appropriate to external loads is feasible. To reach this aim, NiTi wires and a polymer matrix may be co-deposited simultaneously using an Arburg plastic freeformer. The actuator abilities and reliability are largely affected by the load transmission between the NiTi wire and the polymer matrix. Therefore, different surface treatments are investigated to improve the adhesion. The influence of various treatments is quantified by performing pull-out tests and evaluating the surface roughness. The biggest impact on the interfacial shear strength (ISS) was achieved by a laser gas nitration of the wire surface before embedding them in the matrix material. This approach increased the ISS by a factor of 11 compared to the initial state.

1. Introduction

Shape memory alloys (SMA) feature a high specific actuation energy due to fact that they can be largely deformed and then return to their initial shape by heating. Several applications can be found in literature, especially for automotive [1], aerospace [2] and medical [3] purposes. In the aforementioned applications, NiTi is mainly used as bulk material. Additive manufacturing (AM) has been used to produce actuators using shape memory polymers (SMP). Specimens made from SMP can be easily linked to other thermoplastics, but have to be activated by heat from the outside which makes them react slowly [4,5]. Actuators made from SMA wires integrated in polymer matrices using AM, react much faster since actuation via Joule heating is possible. Producing actuators this way, offers a high design freedom and results in many benefits including actuator function, vibration control, damping, sensing and self-healing [6,7]. To consider the potential of AM and to manufacture parts with complex shapes, it is necessary to integrate the SMA wires during the printing process. For the integration of reinforcing fibres into functional parts during additive manufacturing processes, several approaches can be found in literature [8–10]. For instance, a machine module for the Arburg freeformer has been developed capable of integrating endless carbon fibres or glass fibres into manufactured components locally [9,11]. Due to the geometric similarity of reinforcing fibres and the SMA wires, this process shows great potential for the automated integration of SMA wires. For efficient actuator function, strong interfaces between matrix material and NiTi wire are necessary [12]. The interfacial adhesion can be improved by different mechanical, thermal and chemical treatments, which mostly aim to increase the surface roughness of the NiTi wire [13]. To compare the effect of different surface treatments pull-out or push-out tests can be used to determine the interfacial shear strengths (ISS). This allows the evaluation of the effect achieved by the treatment independently from the diameter and the embedded lengths of the wire.

Merlin et al. were able to increase the ISS by 50 % performing chemical etching of the NiTi wire surface in a 40 % solution of HNO_3 for 30 min. The chemical etching produced oxide layers which increased the surface roughness and therefore enhanced the bonding between matrix material, in this case a polyester resin, and wire surface [13]. Furthermore, oxidation of the wire surface induces the formation of titanium oxides on the wire surface and corresponding hydroxyl groups (- OH) enable bonding to polymers forming hydrogen bonds [14,15].

Neuking et al. used polyamide (PA 6) as matrix material and were able to improve the ISS by coating the NiTi-wires with a solution of PA 6 (Ultramid 1C), ethanol and water before embedding them into the PA 6 matrix using injection moulding. Coating the wires, the ISS could be increased by 400 % to 4.5 ± 0.5 MPa [16].

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Man and Zhao created TiN dendrites using a laser gas nitriding process (LGN)[17]. A continuous wave 2 kW Nd-YAG Laser was used to irradiate the NiTi surface, while high purity nitrogen gas was discharged to the surface at the same time. A three-dimensional dendritic network was created, which increased the surface area for mechanical anchoring. LGN was performed on NiTi plates and two plates were overlapped respectively and joined in an area of 6 × 7 mm with a two-component epoxy resin as the adhesive (lap joint samples). Compared to untreated samples, the increase in adhesive strength was above 150 % and a shear stress of about 15 MPa was achieved. Etching of the LGN surface in a solution of 10 % HF and 40 % HNO_3 for 30 min, increased the shear strength to about 20 MPa [17].

2. Experimental

In the study at hand, the influence of different surface treatments was investigated by means of pull-out tests. Based on the results in the literature and preliminary investigations, different surface treatments were carried out on NiTi wires, which were then embedded in polymer matrices using additive manufacturing in a modified FDM process patented by Arburg GmbH [11]. Maximum pull-out force was determined to determine the interfacial shear strength.

2.1. Materials

The wires considered within this study were provided by G. Rau GmbH & Co. KG in Pforzheim, Germany. They consist of a NiTi alloy with 49.7 at% Nickel and featured a diameter of 80 ± 1 µm. To determine the effect of surface treatments, the NiTi wires were heated up to 100 °C in a furnace for one hour to restore their original shape. Afterwards they were cleaned in an ultrasonic bath with acetone. Different surface treatments, as described in section 3.3, were performed. All surface treatments were examined using a Keyence laser scanning confocal microscope and the surface roughness R_a was measured likewise. R_a signifies the arithmetical mean deviation of the assessed profile. In a next step, the wires were embedded in an acrylonitrile butadiene styrene (ABS, Ineos Styrolution Terluran GP-35) matrix as shown in Fig. 1 using the Arburg freeforming process which is described in detail in section 3.2. Glass transition temperature of the matrix material is about 95 °C, which was determined using a differential scanning calorimetry (Netzsch DSC 214 Polyma). Likewise, the transition temperatures of the NiTi alloy were determined which are stated in Table 1.

Table 1
Transformation temperatures of the NiTi wire determined by differential scanning calorimetry.

Parameter	Value
Martensite start temperature (M_s)	31.5 °C
Martensite finish temperature (M_f)	24.6 °C
Austenite start temperature (A_s)	71.1 °C
Austenite finish temperature (A_f)	84.8 °C

2.2. Manufacturing process and printing of the specimens

In the study at hand, the Arburg plastic freeforming (APF) process was considered to manufacture the pull-out specimens. One major advantage of this manufacturing process is the usage of standard granulates from injection moulding. This allows a greater variety of usable materials compared to other additive manufacturing processes [18].

In the first step of the APF process (cf. Fig. 2) the plastic granulate (1) is drawn into the extruder screw (2), where it is heated, homogenized, and pressurized before entering a nozzle assembly (4). A piezo actuator (3) is installed in the nozzle, which opens and closes the nozzle at a high frequency. This way, individual droplets are discharged and deposited next to each other layer by layer [18].

Similar to other additive manufacturing processes, there are small gaps between different droplets of the material. To transfer the load between the wire and the matrix, it is necessary to obtain a polymeric matrix with as little voids as possible. For this purpose, a determination of the manufacturing parameters for the used materials is necessary. During this material qualification, various machine parameters and slicing parameters need to be determined. The discharge coefficient is the most important machine parameter for high-quality components and refers to the volume of a single drop of discharged polymer. This volume is then used to define the slicing parameters for this material. The distance (d_d) between individual drops and the individual layer height (lh) of the component are important for the AM process (cf. Fig. 3).

In general, it should be noted that droplets form no perfect spheres when discharged. During discharge the polymer still has a very low viscosity and thus adapts to the previously discharged drops and the layer underneath. As a result, filling degrees far above the theoretically densest spherical packing (≈74%) are achieved. In further trials, the optimum filling degree, i.e. the lowest possible void content, is determined by iteratively varying the distance between the droplets during slicing, while maintaining dimensional accuracy. For this purpose, the dimensions of the components are measured with a caliper gauge and the density of the component is determined using the Archimedes method and compared to the density of the polymer granulate. Hence, the filling degree of the component is calculated.

The following process parameters and the filling degree (cf. Table 2) were determined for printing the specimens with ABS, achieving minimal void content while retaining dimensional accuracy.

The shape of the specimen is shown in Fig. 1. With a height of 4 mm it consists of 20 polymer-layers with a layer height of 0.2 mm. At the tenth layer the AM-Process was paused and the SMA-wire was integrated and fixed manually. After a few seconds, the printing process was restarted and the ten upper layers were fabricated to finish the pull-out specimen. For the study at hand, machine paths have been created manually. An automated insertion of the wire for future prospects is subject of the current research. Regarding a local and automated integration of the wires it will be necessary to adjust the process parameters.

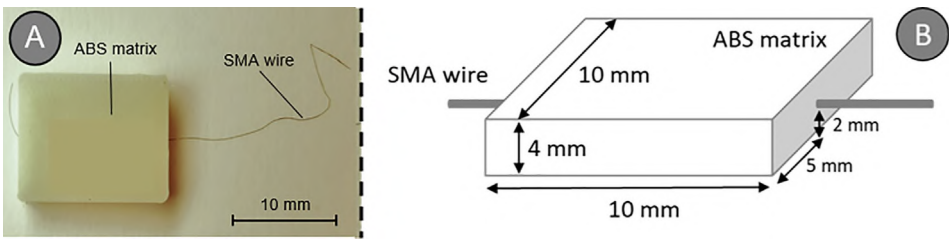

Fig. 1. Pull-out sample produced from a NiTi wire embedded in an ABS matrix (A); specimen dimensions (B).

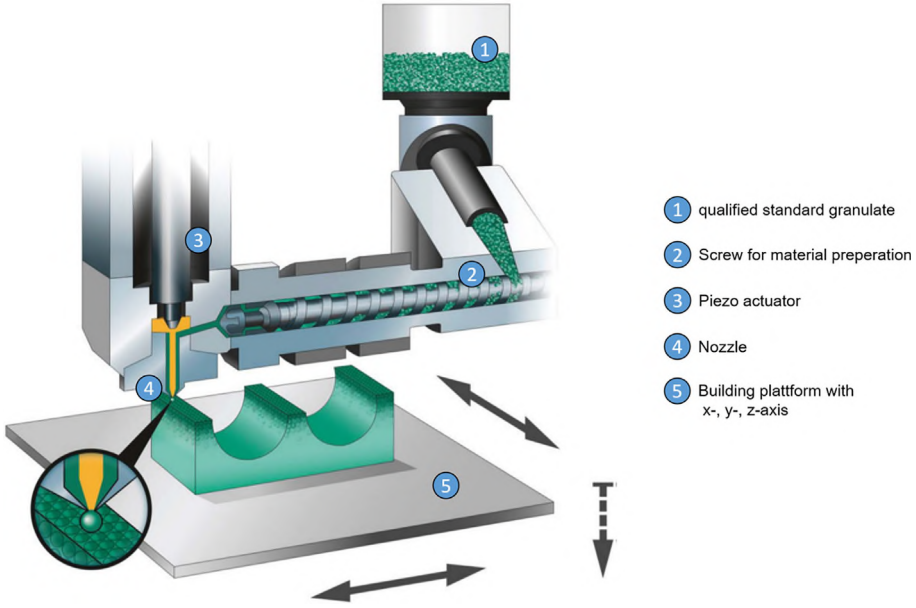

Fig. 2. Arburg plastic freeforming (APF), according to [18].

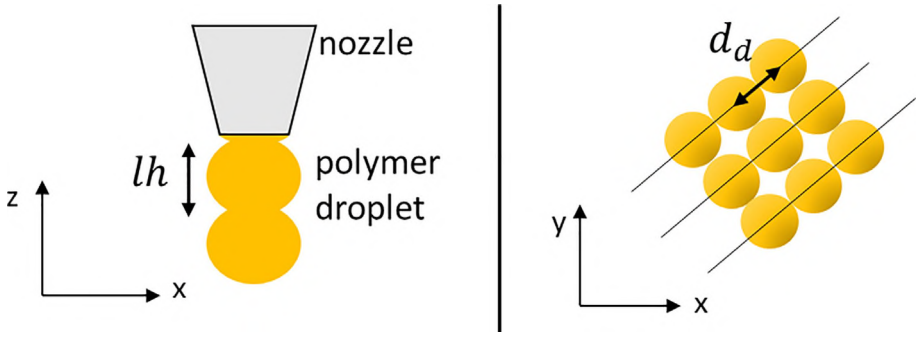

Fig. 3. Layer height (*lh*) and the distance between droplets (d_d), based on [18].

Table 2
Parameters for the sample manufacturing using the Arburg plastic freeforming process.

Parameter	Value
Discharge coefficient	72 %
Nozzle temperature	270 °C
Chamber temperature	80 °C
Layer height	0.2 mm
Shape factor (d_d/lh)	1.34
Filling degree	96.4 %

Table 3
Parameters of the different surface treatments conducted.

Surface treatment	Parameter	Dwell time
Initial state	–	–
Etched	40 % HNO_3	30 min
Etched	40 % HNO_3	8 h
Oxidised	500 °C, 20 % O_2 + 80 % N_2	2 h
Oxidised	500 °C, 20 % O_2 + 80 % N_2	5 h
ABS coated	ABS granulate dissolved in Acetone	2 h
LGN	Power intensity: 6 W frequency: 50 kHz, scan speed: 100 mm/s, spot size: 40 µm, scan line spacing: 30 µm, N_2 gas flow rate: 4/70 L/min	5 min

2.3. Surface treatments of the NiTi wires

Based on the results in the literature and preliminary investigations, different surface treatments were selected for this study in order to increase the adhesion between the wire and the polymer matrix. All relevant parameters are listed in Table 3.

2.3.1. Untreated (reference)

As reference, untreated wires, cleaned in an ultrasonic bath with acetone for 5 min, were considered. The wire surface is shown in Fig. 4a.

2.3.2. Etched surface

The wire was cleaned in ethanol in an ultrasonic bath and etched in 40 % nitric acid afterwards. Some wires were etched for 30 min and some for 8 h to investigate the influence of the etching time on the adhesion between wire and polymer matrix. The wires were air dried before they were embedded in the ABS matrix. Fig. 4b shows a photomicrograph of the etched surface.

2.3.3. Oxidised surface

The wire was heated to 500 °C in a tube furnace with artificial air atmosphere (20 % O_2 and 80 % N_2) to encourage oxidation of the wire

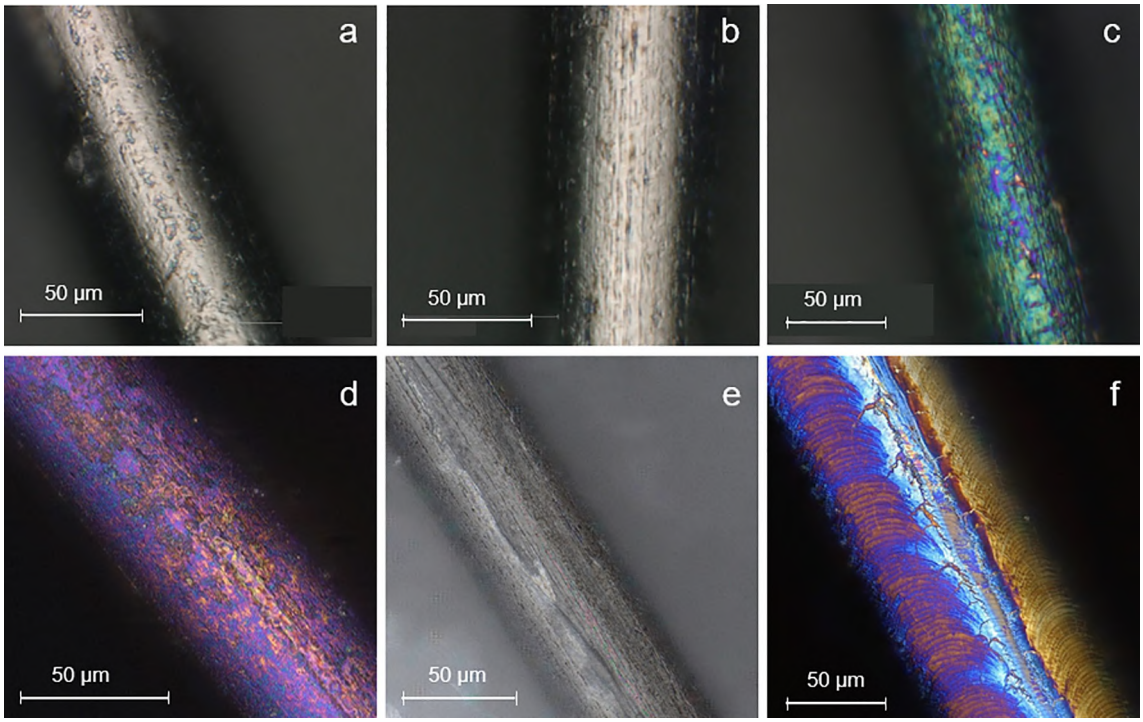

Fig. 4. Microscopic images of the surface treatments: a) untreated, b) etched, c) oxidised for 2 h, d) oxidised for 5 h, e) ABS coated, f) laser gas nitrified.

surface. One wire was kept in the furnace for one hour, another one for five hours. Microscope images of the surface oxidation are shown in Fig. 4c and Fig. 4d.

2.3.4. ABS coated

ABS granulate (Terluran GP-35) and acetone were mixed in a weight ratio of 1:4 at 60 °C in a magnetic stirrer until there was a homogenous dissolution. Afterwards, the NiTi wire was soaked in the dissolution for 2 h at 60 °C. Then, the wire was air dried before it was embedded in the ABS matrix. Fig. 4e shows a photomicrograph of the ABS-coated surface.

2.3.5. Laser-Gas-Nitration (LGN)

LGN of the surface aimed to increase the surface roughness of the wire and to improve the interlocking between the wire and the polymeric matrix material. Therefore, a 30 W fibre laser (Gie-tec gie-MARK30++) with a wave length of 1064 nm and a spot size of 40 µm was used at 20 % power intensity. The frequency was set to 50 kHz, the scan velocity was set to 100 mm/s and the scan line spacing was set to 30 µm. In order to achieve an equable nitration of the whole surface, the wire was scanned from both sides, using a specific mount, designed for this purpose. During the laser treatment the wire was overflown with nitrogen gas with a flow rate of 4 L/minute to initiate a nitration process at the point where the laser fused the surface. Fig. 4f shows a photomicrograph of the laser gas nitrified surface. One wire was embedded in the polymer matrix after LGN directly, whereas another one was cleaned with isopropanol before embedding in the polymer matrix.

Due to the good preliminary results of the samples manufactured with these wires (cf. chapter 4), the mount system was optimised in order to realise laser treatment on the whole wire surface and the gas flow rate was increased to 70 L/minute to achieve a higher homogeneity of the surface nitration. The new mount system consists of two cubes with a hole where the wire is threaded and clamped with screws as it can be seen in Fig. 5. The cubes are placed on the base plate and rotated by 90° after each laser treatment. Likewise, the wire is laser treated on four sides.

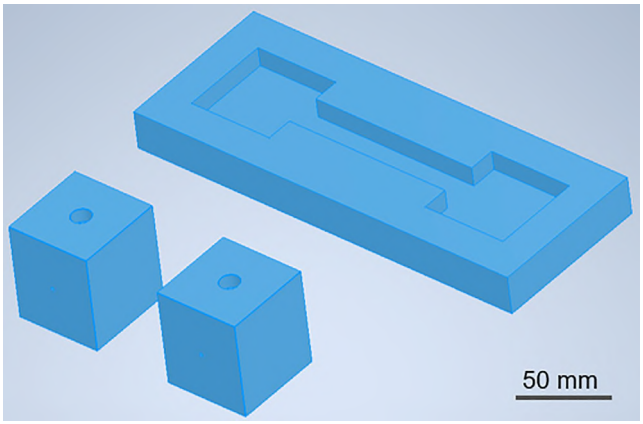

Fig. 5. CAD model of the new mount system. The wire is clamped between the two cubes and is laser treated on four sides by turning the cubes on the base plate.

2.4. Mechanical testing

Pull-out tests were performed on a ZwickRoell zwickiLine 0.5 universal testing machine with a load cell capacity of 5 N. The composite was held by a metal sheet with a hole in the middle, constructed for this purpose (cf. Fig. 6). The metal sheet held the composite at its position without putting compressive forces on the interface while the wire was clamped in the gripping jaw connected to the load cell. The wire was pulled out of the composite with a constant speed of 1 mm/minute, while the tensile force was measured. Displacement of the wire was measured by the displacement of the traverse. For each surface treatment, four samples were tested and the arithmetic mean of the pull-out force as well as the standard deviation were determined.

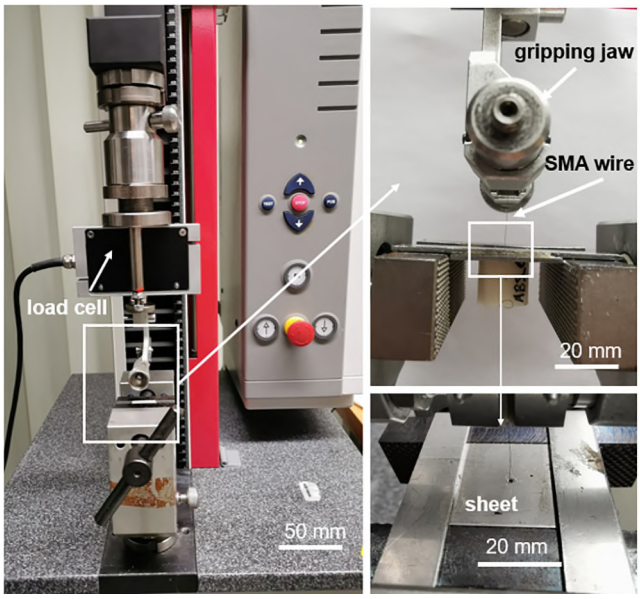

Fig. 6. Set-up for pull-out tests on a ZwickRoell zwickiLine 0.5 with 5 N load cell and mount board for holding the composite sample.

3. Results and discussion

A representative tensile force–displacement-diagram for each surface treatment is shown in Fig. 7. At a first sight, the highest pull-out force was achieved with the laser gas nitrated wire. In order to compare the influence of the different surface treatments independently from the embedded length of the wire, the interfacial shear strength (ISS) is calculated. However, the peak value of the curve in Fig. 7 is probably not the force where the first damage of the interface happens. After this initial damage the force can rapidly oscillate around a mean value or settle down to a constant value due to a stick–-slip behaviour [19]. Therefore, for the calculation of the ISS, the value of the tensile force corresponding to the first dip in the force–displacement curve is considered, as shown in Fig. 7. The ISS is calculated using eq.1, where F is the aforementioned pull-out force, d is the diameter of the wire and l is the length of the embedded wire. Embedded length of the wire was measured individually for every sample using a calliper gauge. The arithmetic mean values as well as the standard deviation of the ISS resulting from different surface treatments are shown in Fig. 8.

$$\tau = \frac{F}{d \cdot \pi \cdot l} \tag{1}$$

3.1. Etched surface

Etching of the wire surface for 30 min had almost no influence on the ISS and could improve the adhesion by 2 % only. This stands in contrast to the results of Merlin et. al. who were able to increase the ISS by 50 % using the same etching procedure [13]. In contrast, etching the wires for 8 h in the study at hand, increased the ISS by a factor of 2 from 0.12 MPa to 0.24 MPa. Likewise, the surface roughness was increased by a factor of 2. Nevertheless, due to the results in the study at hand, other investigated surface treatments are more promising.

3.2. Oxidised surface

Oxidation of the wire surface for two hours increased the ISS by a factor of 3.8 from 0.12 MPa to 0.46 MPa, but the ISS could not be increased any further by leaving the wires in the furnace for five hours. In this case the ISS increased to 0.45 MPa. Apparently, the surface was already fully oxidised after two hours and therefore longer annealing times had no influence on the surface oxidation. The measured surface roughness of the wire, annealed for five hours, could not be increased.

3.3. ABS coated

The ABS coating had a significant impact on the ISS, whereas the standard deviation is comparatively high. The same applies to the surface roughness as specified in Table 4. Fig. 4e shows inhomogeneities of the coating, which could be an explanation for the high standard deviation. Retrospective, the viscosity of the ABS dissolution was probably too high which resulted in these inhomogeneities. ISS could be increased by a factor of 4.4.

3.4. Laser-Gas-Nitration (LGN)

Laser gas nitration of the wire surface using the primary mount system had almost the same impact on the ISS as the ABS coating. ISS could be increased by a factor of 4.1 to a value of 0.49 MPa, which

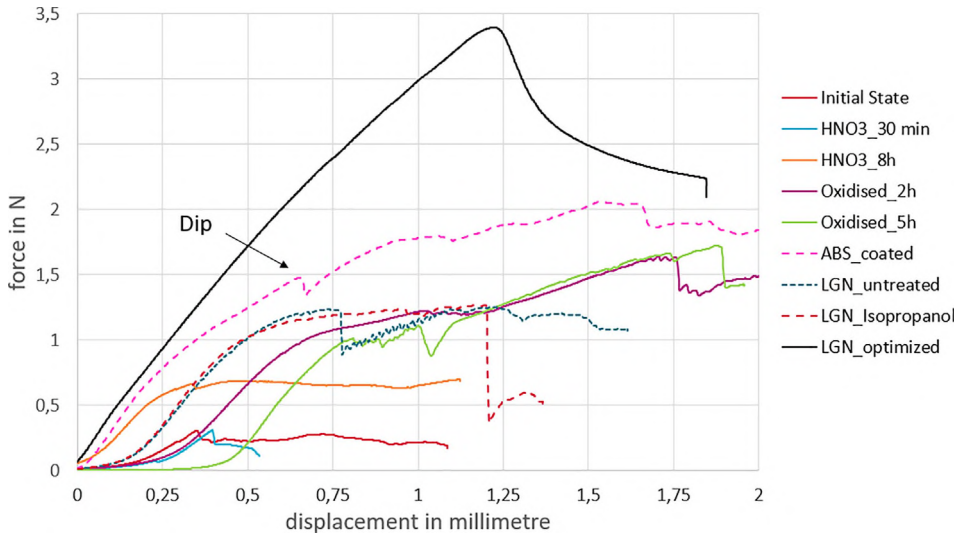

Fig. 7. Exemplary load–displacement curves for every surface treatment.

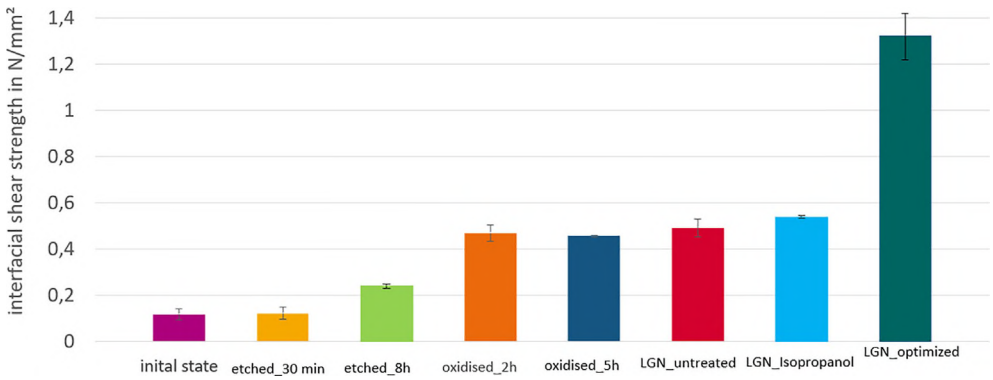

Fig. 8. Average resulting interfacial shear strengths for every surface treatment.

Table 4
Measured surface roughness for every conducted surface treatment.

Surface treatment	Surface roughness R_a in µm
Initial state	0.67 ± 0.08
Etched 30 min	0.74 ± 0.01
Etched 8 h	1.31 ± 0.11
Oxidised 2 h	2.9 ± 0.82
Oxidised 5 h	2.86 ± 0.12
ABS coated	2.19 ± 0.53
LGN untreated	6.14 ± 1.49
LGN Isopropanol	6.32 ± 1.31
LGN optimized	5.28 ± 0.63

is more than the increase Man and Zhao were able to achieve [17]. Nevertheless, the absolute value of the adhesion strength they achieved was ten times higher. As aforementioned in chapter 2, NiTi plates with a size of 6 × 7 mm were used for their investigation. Because of the flat surface, the surface structure they achieved by laser gas nitriding was more homogenous compared to the wire surface achieved in the study at hand. Furthermore, a two-part slow curing epoxy resin was used and a compressive load was applied during the curing stage, which probably increased the adhesion [17]. In the study at hand, ISS and surface roughness could be further increased to 0.53 MPa by cleaning the laser gas nitrified surface with isopropanol before the wire was embedded in the polymer matrix (cf. Table 4). By improving the mount system and increasing the nitrogen gas flow rate to 70 L per minute, the ISS could be increased by a factor of 11.3 to a value of 1.32 MPa compared to the untreated samples and by a factor of 2.7 compared to the LGN with the primary mount system. In contrast, the surface roughness could not be increased any further.

As the surface treatment could change the transition behaviour of the SMA wires, the transition temperatures of all treated wires were determined after treatment using differential scanning calorimetry. The results are listed in Table 5. All performed surface treatments increased transition temperatures, but the phase transition still took place and therefore the actuator function of the SMA is still applicable.

Although the ISS could be increased by almost every type of surface treatment, the adhesion between the NiTi wire and the matrix still needs to be increased for an appropriate actuator function. Adhesion is not only influenced by different surface treatments, but also by the manufacturing process [20]. Paine et. al. stated that the adhesion could be increased by heating the wires while embedding them into the polymer matrix to improve the interlocking [19]. Temperatures of the wire above the glass transition temperature (T_g) of the matrix polymer in use are preferable. Although, in this case the temperature of the wire must stay below the austenite start temperature (A_s) to prevent phase transformation. Phase transformation during the overprinting of the wire would cause recovery of the wire to its original shape and prevents shape deformation of the composite by heating the wire. Therefore, phase transformation during the overprinting would cause loss of the actuator function. In the study at hand T_g of ABS is about 92° C, whereas the A_s temperature is dependent on the surface treatment as stated in Table 5. One opportunity to increase the transformation temperatures, particularly the A_s temperature, is to apply tensile strengths to the wire while overprinting them. Investigations as part of a different study showed, that the A_s temperature could be increased up to 150 °C by applying a tensile strength of 500 °MPa to the wire.

Another possibility to increase the adhesion would be a reduction of the viscosity of the polymer droplets during the overprinting process. This would be reached by increasing the temperature at the nozzle. As aforementioned, the temperature of the wire must stay below A_s , which is the limiting factor due to the heat input from the molten polymer droplets into the wire. In a previous work, it was shown that changes in the mechanical properties of fibre-reinforced polymeric samples were achieved by varying the process parameters of the APF, for example the nozzle temperature and the chamber temperature [8]. Therefore, an investigation of the manufacturing parameters

Table 5
Transformation temperatures of the pre-treated NiTi wires determined by differential scanning calorimetry.

Surface treatment	Martensite start temperature M_s	Martensite finish temperature M_f	Austenite start temperature A_s	Austenite finish temperature A_f
Initial state	31 °C	24 °C	71 °C	85 °C
Etched 30 min	40 °C	35 °C	69 °C	85 °C
Etched 8 h	42 °C	27 °C	67 °C	86 °C
Oxidised 2 h	66 °C	55 °C	83 °C	100 °C
Oxidised 5 h	65 °C	58 °C	80 °C	97 °C
ABS coated	55 °C	35 °C	66 °C	86 °C
LGN	67 °C	56 °C	70 °C	94 °C
LGN + Isopr.	65 °C	53 °C	73 °C	97 °C
LGN optim.	60 °C	40 °C	68 °C	86 °C

for composites of polymers and SMA wires should be done to further improve the adhesion.

Nonetheless, LGN of the wire surface and embedding them into a polymer matrix using the APF process is a promising approach for the manufacturing of actuator parts. Due to the high design freedom of the APF process, actuators can be individually designed for each purpose and actuation energy can be individually adjusted by varying number and position of the SMA wires in the polymer part. High specific actuation energy and functionality independent from power supply predestine these shape memory actuators for remote places with limited space as it is the case in turbines for example. Shape memory actuators can be used for a variation of the chevron geometry in order to reduce noise production during take-off [21].

4. Conclusion

Different surface treatments were investigated to improve the adhesion between a NiTi wire and an ABS polymer matrix. The following surface treatments were performed on the wire surface: etching with HNO_3 for 30 min ad for eight hours, oxidation at 500 °C for two hours and for five hours in oxygen atmosphere, ABS coating and laser gas nitration. The wires were embedded in an ABS matrix using the Arburg plastic freeforming process. Adhesion was characterized by performing pull-out tests to determine the interfacial shear strength (ISS). The greatest increase of ISS was achieved by the optimized laser gas nitration of the wire surface. Thus, the ISS could be increased by a factor of 11.3. ABS coating of the wire surface increased the ISS by a factor of 4.4, whereas oxidation of the wire surface led to an increase of the ISS by a factor of 3.8. By etching the wire surface, an increase of the ISS by a factor of 2 was achieved. Nevertheless, the adhesion between NiTi wire and polymer matrix must be further increased for an appropriate actuator function. The LGN process needs to be further improved to reduce the variation of the surface quality as it is one of the most promising surface treatments to increase the adhesion. Furthermore, the adhesion could be enhanced by combining the LGN and the ABS coating of the wires. Above all, the manufacturing process must be improved performing a study of the process parameter to increase the adhesion between wire and polymer.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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