A prototype of a new shape-memory nitinol knitted fabric intended for use as an active thermal insulating interlining in firefighting protective clothing was developed in the study presented in this paper. Weft knitted fabrics were made from commercially available cold-worked nickel titanium alloy monofilaments. Knits were made on a manual knitting machine from a monofil measuring 0.1 mm in diameter, while a hand-made knit was prepared from a monofil measuring 0.2 mm in diameter. Nitinol fabrics were annealed at 500 °C to achieve an austenite transition temperature of 75 °C. A special constructed mould made of a steel frame and aluminium domes measuring 30 and 20 mm in height was used to give the nitinol fabrics a new temporary shape. A two-way, shape-memory effect of the nitinol fabrics was achieved using a 15-cycle training process. The achieved shape-memory effect was tested in a heated chamber at 100 °C, where bulges measuring 12–25 mm in height occurred. NiTi knits made from finer monofil were the most successful shape-memory knits. They were machine knitted and achieved sufficiently high bulges, measuring 18 or 12 mm, that facilitated large enough air gaps for effective thermal protection. A smart textile system was prepared by inserting the trained nitinol fabric into a pocket made from two textile fabric layers sewn together. When it was exposed to environmental temperatures of 75 °C and higher, it instantly changed its form from a two-dimensional shape to a three-dimensional shape, while increasing the air gap in the pocket. A quilted fabric made from such a smart textile system could be used in firefighting protective clothing to locally improve thermal insulation and protect the human skin from overheating or burns.

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(Some figures may appear in colour only in the online journal)
stockings and bandages [6, 7], more flexible and adaptable bra bones for underwire bras, seatbelts that increase the wearer’s safety in car crashes [8], improved cushioning pads [9], etc. For the most part, shape-memory garments with nitinol incorporated into woven or knitted fabrics facilitate shape recovery through human body heat or heat during ironing.

Amongst shape-memory materials, shape memory alloys are characterised by large deformations induced and recovered through changes in temperature or stress [10]. Amongst shape memory alloys, NiTi alloy is the first choice on account of its high performance, bio-compatible material [11]. NiTi alloy is a thermo-responsive material [11] with thermally triggered microstructural transformations from a martensite state to an austenite state, and vice versa, in a temperature range from \(-100 \, ^\circ \text{C}\) to \(+100 \, ^\circ \text{C}\) [12], depending on the ratio of nickel and titanium in the alloy, and on the annealing temperature of the cold-worked alloy. In a pure austenite state at high-temperatures, NiTi alloy is hard and rigid, but at low-temperatures, when the alloy is in a pure martensite state, it is soft and flexible. NiTi alloy monofilons can therefore be integrated into textile fabrics using traditional techniques, such as weaving, knitting, stitching and braiding at room temperature, similar to textile yarns. Textile fabrics containing NiTi alloy monofilons are expected to improve the aesthetic [13], anti-static [14], shielding [15] and electroconductive [16] functions of textiles, similar to other metallic fibres made from gold, silver, copper, stainless steel, etc. The most prominent metallic fibre properties are cross-section (round, elliptical or square) and thickness, which usually ranges from 1 \(\mu\text{m}\) up to 80 \(\mu\text{m}\) [17]. Coarser metallic fibres are stiffer and can substantially reduce fabric flexibility, drape and handle, especially when the proportion of metal fibres is high [18].

NiTi monofilons could be embedded in textiles in so-called pre-annealed or annealed forms. When using a pre-annealed NiTi alloy, fabrics with embedded NiTi alloy monofilons should be trained to achieve a shape-memory effect. The training temperatures usually do not exceed 100\(^\circ\text{C}\), which facilitates the use of NiTi alloy in almost all fabrics from organic fibres. The disadvantage of using a pre-annealed NiTi alloy lies in the limited supply of monofilons of the desired thickness and appropriate transition temperatures on the market, and the high prices thereof.

Annealed NiTi monofilons are already trained and usually take a flat temporary form. Their use is similarly limited, as is the case with pre-annealed NiTi monofilons, but they could be successfully used exclusively in woven fabrics.

Annealing cold-worked NiTi monofilons at a selected temperature in a range between 400\(^\circ\text{C}\) and 600\(^\circ\text{C}\) [1] facilitates the setting of the desired transition temperatures of crystal structure transformation from a martensite state to an austenite state and vice versa: i.e. martensite start \((M_s)\) and martensite finish \((M_f)\) temperature, and austenite start \((A_s)\) and austenite finish \((A_f)\) transformations. Annealed NiTi monofilons must be shape-memory trained to recall their temporary shape. A high-temperature annealing process limits the use of NiTi monofilons in fabrics made from 100% NiTi monofilons and in combinations only with high-temperature resistant fibres, such as glass, basalt or carbon fibres [19]. Thus, cold-worked NiTi monofilons are particularly useful for functional textiles because they facilitate the setting of transition temperatures with respect to a specific use.

Presented below are examples of the use of NiTi monofilons in smart firefighting protective clothing with active thermal regulation. This was achieved by adapting the thickness of the air gap between the fabric layers in clothing.

An air gap is the empty space between the skin and a fabric, or between two fabrics in an article of clothing [20]. When the air captured in the air gap is stationary, it increases the thermal insulation of clothing, provided that the textile materials are dry. Song [21] found that an air gap of 7–8 mm between the skin of the human body and a single-layer protective garment facilitates the most effective thermal insulation under flash fire conditions. He also proved that the increased thickness of the air gap is only effective when convection, which worsens the thermal insulation of a garment, is prevented. Russell et al found that an air gap between two low-air permeable fabrics effectively increases the thermal insulation of clothing [22].

In patent no. WO 97/42026 from 1997, Russell et al [22] explained the idea of using NiTi alloy for thermally insulating textiles with a variable air gap that provides user-suitable thermal protection. They attached NiTi monofilons in a grid pattern on a textile substrate, and then inserted it between two fabric layers. The NiTi alloy layer was thin at room temperature, but wrinkled and increased the air gap between fabric layers when the temperature of the environment was increased up to 70\(^\circ\text{C}\). This concept is associated with the thermal insulation concept of increasing the number of clothing items, where the amount of air between clothing layers and the air captured inside the clothing increase with the number of layers [22]. This concept is effective at very low and at higher temperatures.

Congalton published a completely new solution for thermal insulation at higher environmental temperatures, where he presented a smart thermal insulation jacket with incorporated conical springs made from a NiTi monofil with a diameter of 1 mm and an actuation temperature of around 50\(^\circ\text{C}\) [23]. Springs could be easily inserted into a double-fabric garment by attaching one end of a spring to the inner fabric and the other end of the spring to another fabric. According to information presented in the paper, a maximum air gap of 35 mm was required for effective protection against burns. He also predicted that such panels with air gaps would only be sufficient on strategic areas of the body.

Yoo et al made a smart insulating jacket for protection in extreme temperature conditions by incorporating NiTi alloy monofilons in the form of helical coils [24]. The coils increased the air gap when the environmental temperature changed and reached the NiTi alloy coils’ pre-programmed transition temperature. Changes in the shape of the entire jacket depended on the number of NiTi alloy coils integrated into the textile fabric and on the fabric construction, i.e. its possibility of deformation.

Michalak and Krucinska [25] prepared active spiral elements from a NiTi monofil with a diameter of 0.18 mm for
use in a textile layered structure to adapt its thermal insulation to a hot environment. The spirals had a two-way shape-memory effect: they contracted when heated and expanded at lower temperatures. This smart textile system incorporated into clothing could effectively develop a maximum air gap of 7.5 mm in a cold environment and a minimum air gap of 1.75 mm in a hot environment.

According to Congalton’s study, the human body suffered second degree skin burns after 38 s in a standard protective garment exposed to a heat flux of 15 kW m⁻²; when SMA springs with an austenite temperature of 57 °C were incorporated into the garment, that time was prolonged to 50 s [23]. It could be concluded that a suitable transition temperature of NiTi knit for use in heat protective clothing is around 60 °C.

The aim of our study was to develop a smart interlining for firefighting protecting clothing that would provide dynamic thermal isolation properties. Instead of using the spirals referred to in other studies, [17–22] we developed a prototype of a NiTi alloy knitted fabric with a two-way shape-memory effect that facilitates increasing thickness at a temperature of 75 °C, and higher and decreasing thickness when the environmental temperature falls below 75 °C.

2. Experimental

2.1. Material

Cold-worked NiTi alloy monofilts were used to produce smart textile material (table 1). Their nominal composition and transition austenite temperature range provided by the manufacturer are presented in table 1.

The stress/strain curves presented in figure 1 were calculated from force/elongation data collected in a tensile test of cold-worked NiTi monofilts on an Instron 5567 dynamometer, using specially designed Instron 2714 series pneumatic grips that provide a gradual increase in gripping force over a polished and specially contoured surface. The test was performed at a gage length of 250 mm and speed of 5 mm min⁻¹.

![Figure 1](image)

**Figure 1.** Stress–strain curves of used cold-worked monofilts NiTi#6 (0.1) and NiTi#6 (0.2), measured on an Instron 5567 dynamometer at 20 °C.

| Properties                  | Monofil NiTi#6 (0.1) | Monofil NiTi#6 (0.2) |
|------------------------------|----------------------|----------------------|
| Diameter (mm)                | 0.1001               | 0.2200               |
| Nominal composition of NiTi (wt%) | 55.47/44.53          | 55.47/44.53          |
| A₁ (°C)                      | +40 up to +80        | +40 up to +80        |

2.2. Determination of NiTi alloy transition temperatures

In order to set the desired transition temperatures, the cold-worked NiTi#6 (0.1) and NiTi#6 (0.2) monofilts were annealed for 30 min at three different temperatures, between 400 °C and 500 °C, followed by cooling at an air temperature of 22 °C.

To determine the transition temperatures of annealed NiTi alloy, a differential scanning calorimetry (DSC) method was used. Measurements of the transformation temperatures—Mₛ, Mₚ, Aₛ, A₁ and the maximum and minimum transformation temperatures (Mₚ, Aₚ)—were made using a Mettler DSC 1 device (Mettler Toledo). Specimens for DSC analysis were prepared as rolls with a diameter of 5 mm and a weight of 30–42 mg. The tests were performed in a temperature range of −50 °C to 100 °C at a heating/cooling rate of 5 K min⁻¹.

2.3. Knitting of NiTi fabrics

A manual Singer Superba knitting machine was used. All the machine-knitted samples were made using the same machine parameters. Needles with a thickness of 3.25 mm were used for hand knitting.

2.4. Annealing and shape-memory training of NiTi knits

A specially designed mould for annealing and training the NiTi knits was made (figure 2). The frame (figure 2, right) and the basic plate (figure 2, left) were made of stainless steel, while the domes fixed on the basic plates were made from aluminium alloy. The height of the bigger dome was 30 mm, while the height of the smaller two domes was 20 mm.

Before annealing, a NiTi knit was inserted into the mould frame. It was annealed in a flat state at a temperature of 500 °C for 30 min. The NiTi knit was then shape-memory trained using the following cyclic temperature training method (figure 3):

1. Annealed NiTi knit was air cooled in a flat state at a temperature below Mₛ = 22 °C for 20 min (figure 3, step 1).
2. The NiTi knit was inserted in the mould to set the desired shape of a dome (figure 3, step 2).
3. The NiTi knit, clamped in the mould, was heated to a temperature above A₁ = 75 °C for 10 min (figure 3, step 3).
To obtain a stable two-way memory effect, steps 1–3 were repeated 15 times.

2.5. Compression test

A compression test of bulges was performed on the NiTi knits in an austenite state using an Instron universal electronic dynamometer (5567 series). The test was performed in a heat chamber at a temperature of 100 °C at a deformation speed of 5 mm s⁻¹. The diameter of the compression plate was 58.75 mm and stopped few millimetres from the round table on which the knit was placed.

3. Results and discussion

Preparing the NiTi monofil for training and knitting, making the NiTi knits, the shape-memory training of the knits and the application thereof were very demanding processes that required interdisciplinary knowledge.

3.1. Mechanical properties

It is evident from the stress–strain curves presented in figure 1 that the yield strength of the cold-worked NiTi monofilms measured at room temperature was 1340 MPa for NiTi#6 (0.1) and 1454 MPa for NiTi#6 (0.2). The tensile strength of the cold-worked monofil NiTi#6 (0.1) was 1693 MPa at strain of 4.6%, and was 1762 MPa at a strain of 5.2% for the monofil NiTi#6 (0.2).

3.2. Transition temperatures

Annealed cold-worked NiTi monofilms in the temperature range 400 °C–500 °C demonstrated transition temperatures $A_t$ of between 75 °C and 67 °C (table 2). According to the purpose of our study to achieve a two-way shape-memory effect of NiTi knits, we had to accept a limitation of the chosen material and set its transition temperature at $A_t = 75$ °C in order to maintain the temperature of $M_t$ at room temperature.

The transition temperatures of annealed NiTi monofilts at 500 °C are clearly visible on a DSC graph (figure 4).

When the NiTi monofil was cooled to a temperature of $−50$ °C and then heated to a temperature of 100 °C, the
transition from a low-symmetric monoclinic B19′ phase to an austenite cubic B2 phase, started at a temperature of 62 °C and reached its peak at 69 °C. The transition to an austenite state was complete at a temperature of 75 °C. The sample in an austenite state was then cooled at the same rate as it was heated (5 °C min⁻¹). At temperatures between 50 °C and 40 °C, it transformed into a so-called R-phase, which was then transformed into a martensite state at temperatures between 32 °C and 22 °C. This transformation reached its maximum at a temperature of 27 °C. At a temperature of 22 °C, the transformation from an austenite state to a martensite state was complete.

After analysing the annealed monofilts on a dynamic scanning calorimetry device, the monofilts with the desired transitions temperatures were selected for further study.

### 3.3. Knitting process

Knitting on a manual Singer knitting machine was successful for knits made from finer and flexible NiTi monofil with a diameter of 0.1 mm. The monofil was unwound from a cylindrical tube to prevent the torsional movement of the monofil. The machine knitting process was slow to avoid the frictional heating of the NiTi monofil and maintained the monofil in a flexible martensite state while shaping it into loops.

Machine knitting using a monofil with a diameter of 0.2 mm caused too many breaks of the needles. It was thus not possible to make a quality sample. After several unsuccessful attempts, the No. 3 knit was made by hand. Due to the hardness of the NiTi monofil, the needles frequently twisted.

### 3.4. Characterisation of knitted samples

NiTi knitted samples (NiTi knits) were made:

1. From a single cold-worked filament and
2. From double-wound, cold-worked filaments.

NiTi knits designated as No. 1a, No. 1b and No. 2 were made on a knitting machine from the same NiTi monofil with a diameter of 0.1 mm, while sample No. 3 was handmade from a NiTi monofil with a diameter of 0.2 mm.

The basic properties of NiTi knits are presented in Table 3. The mass per unit areas of NiTi knits No. 1a and No. 1b differ by only 0.8%. The samples are of equal quality.

Doubling the diameter of the monofil (from 0.1 to 0.2 mm) resulted in a 381.1% increase in mass per unit area. Doubling the NiTi monofilts during knitting (using two folded monofilts) resulted in a larger piece of fabric (No. 2) with a

![DSC thermograph of NiTi#6 monofilts, annealed at 500 °C for 30 min.](image)

**Figure 4.** DSC thermograph of NiTi#6 monofilts, annealed at 500 °C for 30 min.

| NiTi knits | Monofil NiTi#6 | Width x length (cm) | Wales per unit length (wales cm⁻¹) | Courses per unit length (courses cm⁻¹) | Mass per unit area (gm⁻²) |
|------------|---------------|---------------------|-----------------------------------|---------------------------------------|--------------------------|
| No. 1a     | 0.1           | 21 × 18             | 4.76                              | 2.67                                  | 89.15                    |
| No. 1b     | 0.1           | 20 × 17             | 4.80                              | 2.59                                  | 89.85                    |
| No. 2      | 2 × 0.1       | 23 × 21             | 4.35                              | 2.29                                  | 141.45                   |
| No. 3      | 0.2           | 20 × 20             | 5.00                              | 2.40                                  | 339.75                   |

Table 3. NiTi knits properties.
mass per unit area that was 58.04% higher than when one NiTi monofil (No. 1) was used. The number of wales and courses of NiTi knit No. 2 was lower than that of NiTi knit No. 1; because two monofil needed more space, larger loops were formed than in the case of a single monofil.

All the NiTi knits were weft knitted fabrics with a $1 \times 1$ rib-stitch pattern because they do not turn up at the edges.
They could be easily stretched in both directions. Stretching the knits longitudinally caused shrinking in a transverse direction and vice versa [14].

Machine knitted NiTi knits had a regular structure with even courses and wales (figures 5(a) and (b)). The structure of the handmade NiTi knit was uneven with different large loops, which resulted in unevenly formed columns and rows (figure 5(c)).

3.5. Two-way shape-memory effect
NiTi knits No. 1a, No. 2 and No. 3 were trained using a dome with a height of 30 mm to compare their ability to achieve a two-way memory effect. NiTi knit No. 1b was trained using two smaller domes with a height of 20 mm to compare its two-way memory effect with NiTi knit No. 1a trained with a higher dome. Figure 6 illustrates the two-way memory effect achieved on the NiTi knits samples.

The upper images in figure 6 show the NiTi knits in a soft martensite state at room temperature \((T < M_f)\); the middle images show the NiTi knits in a rigid austenite state at a temperature above 75°C \((T > A_f)\); the bottom images show how the NiTi knits returned to a martensite state when the temperature of the material dropped below 22°C \((T < M_f)\). When the NiTi knits were in a soft martensite state, the bulges disappeared, but were not completely flat.

Figure 7 presents the heights of the domes used for training the NiTi knits and the associated bulges created on the NiTi knits when they were in an austenite state. It is evident that the highest bulge of 25 mm was achieved on NiTi knit No. 3 made from thicker monofil. Using two monofil (NiTi knit No. 2) did not result in a height increase; on the contrary, the height was actually lower than when a single monofil (NiTi knit No. 1a) was used. The reasons lie in the greater rigidity of the NiTi knit and higher friction in loops between the two NiTi monofil.

The bulges of both NiTi knits made from a monofil with a diameter of 0.1 mm (No. 1a and No. 1b) reached 60% of the mould dome height. NiTi knit No. 2 reached only 40% of the mould dome height, while the NiTi knit No. 3 reached 83% of the original dome height.

In terms of the shape change speed, the transition from a martensite state to an austenite state when directly exposed to an air temperature of 100°C was rapid and took 30 s. Metal NiTi knits heated up quickly and responded immediately to high temperature, with the bulge shape changing shape from a flat (2D) to a 3D shape. When the NiTi knits were cooled to a room temperature of 22°C, the transition from an austenite state to a martensite state was very slow and lasted at least 30 min. If the NiTi knits were water cooled, the transformation process was faster. Transformations from a martensite state to an austenite state and back were reversible by changing temperature, meaning that the NiTi knits demonstrated the properties of two-way shape-memory. The heights of the NiTi knit bulges achieved with shape memory training were lower than the mould domes used in the process of training; with a higher number of shape change transformations, they became lower and less prominent.

3.6. Compression test of NiTi knit 3D shapes
The compression force of the NiTi knit bulges was measured to determine how the NiTi knits will behave when used as an interlining between two textile fabrics at extreme temperatures. The NiTi knits were tested at 100°C, where they were in a rigid austenite state.

A maximum force of 1.5 cN was achieved while compressing the NiTi knits No. 1a and No. 2 (figure 8), which
were trained using the same dome with a height of 30 mm, while even the forms of their curves differed. The compression curve of the NiTi knit No. 1a is almost linear, while the NiTi knit No. 2 made from two folded monofil is slightly convex. This demonstrates the higher resistance to deformation of the NiTi knit No. 2. In contrast, the deformation curve of the NiTi knit No. 3 had a slight concave form. This knit was compressed using lower resistance. Because it was made from a thicker monofil, however, the maximum compression force of 50 cN was much higher than that recorded for other samples.

The maximum compression force of the NiTi knit No. 1b was 10.6% lower than that of the NiTi knit No. 1a because its bulge was lower.

3.7. Producing a smart textile system prototype

The schemes on the figure 9 illustrate the envisaged manner of incorporating a NiTi knit into firefighting protective clothing to adapt its air gap thickness with respect to the environmental temperature. The outer shell and the moisture barrier should be made from a material that prevents air convection through them. Photos on the figure 9 present a
prototype of a smart textile system that was produced by inserting a trained sample of the NiTi knit No. 3 between two textile fabric layers sewn together into a pocket. When the prototype was exposed to an environmental temperature of 75 °C and higher, it instantly changed its shape by forming a bulge that increased the air gap in the pocket.

4. Conclusion

The NiTi knits No. 1a and No. 1b were the most successful shape-memory knits. They were machine knitted, and achieved sufficiently high bulges of 18 or 12 mm, allowing for large enough air gaps for effective thermal protection. The general findings of the study are:

– A knitting pattern of 1 × 1 rib stitch facilitates the development of bulges on NiTi knits.
– NiTi knits made from thicker monofilts facilitate higher bulges, which were more resistant to compression than those made from finer monofilts. On the other hand, the knitting process using thinner monofilts is more demanding because of higher monofil rigidity.
– Knitting with two folded NiTi monofilts results in heavier knits. They form bulges with lower heights and are less resistant to compressive force than knits made from a single monofil of the same quality.

The conclusions of this research represent the foundation for further research regarding the use of metal shape-memory materials in smart knits. They open a wide range of possibilities for application in human-friendly clothing, interior design, the automotive industry and other areas. By creating unique smart NiTi knits, we have made a step forward in the use of shape-memory alloys in the field of textiles. To date, a shape-memory monofil was no more than a segment inserted into a textile knit in the majority of the smart-shape changing applications. Our research thus presents a wider range of possible applications and the advantages of a smart knit made solely from SMAs.

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