UDC 621.791.947.2
DOI: 10.15587/1729-4061.2025.348866

DETERMINATION OF THE STRUCTURAL FEATURES OF JOINT MATERIAL OF THE NITINOL WIRES MADE BY LASER WELDING

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The object of the study is the welded joint of thin wires made of nitinol alloy. The problem of ensuring the formation of a joint of wires with a diameter of 0.8 mm made of nitinol alloy was solved based on determining the influence of laser welding modes on structural changes in the material of the weld. Based on the use of scanning electron microscopy, micro-X-ray spectral (EDS) analysis, a study of the properties of the material of the welded joint of the nitinol wires was performed. The joint was obtained by welding with an ytterbium fiber laser. It was confirmed that laser welding in an argon atmosphere is able to ensure the high-quality formation of a welded joint without macrodefects while maintaining the superelasticity of the joint material (within the shape memory effect). Two laser welding modes were used with a constant applied laser radiation energy. The duration of the laser radiation action and the multiplicity of such action were varied. At the same time, structural changes in the weld material, which are caused by the multiplicity of laser heating, involve the formation of enlarged zones of the eutectic TiNi + $TiNi_3$ from metastable nanophases of titanium nickelides. The number of such grains decreases with the depth of the weld. Detection of structural changes and establishment of a decrease in the number of point phase inclusions with an increased nickel content in the weld can be a regulating factor for optimizing the structure of the welded joint material. In the welded joints made, when it is bent at an angle of 30°, the residual deformation does not exceed 10%. The results of the study of the structure of the welded joint of nitinol wires made by laser welding are promising and can be used in the conditions of manufacturing nitinol wire connections by welding medical products

***Keywords:** superelasticity of the nitinol joint, laser welding, shape memory effect, intermetallics, eutectic*

Received 03.10.2025
Received in revised form 11.12.2025
Accepted 19.12.2025
Published

***How to Cite:** Kvasnytskyi, V., Zvorykina, A., Zvorykin, L., Zvorykin, C., Kostin, V., Taranova, T. (2025). Determination of the structural features of joint material of the nitinol wires made by laser welding. Eastern-European Journal of Enterprise Technologies, 6 (12 (138)), 6–16. https://doi.org/10.15587/1729-4061.2025.348866*

1. Introduction

Superelasticity of alloys is inherent in materials for which the shape memory effect is characteristic – the ability to return to the original shape. This effect is due to the austenite-martensite-austenite phase transition in a characteristic temperature range. The essence of superelasticity is that the austenite-martensite thermoelastic transition is initiated by mechanical stresses during deformation of the material. Stress relief causes the reverse martensite-austenite transition and the corresponding reverse deformations due to the elastic energy accumulated by martensite and the restoration of the shape [1]. When implementing the thermally stimulated shape memory effect, the material is in the martensite state, in which, after deformation and subsequent heating, it returns to its previous shape. In this case, a cycle of martensite-austenite-martensite phase transformations occurs. There are versions of multi-step shape changes.

Nickel- and titanium-based alloys are the first and most common representatives of shape memory alloys, which are characterized by high corrosion resistance. This determines their use in transplantation medicine [2].

At present, the shape memory effect in titanium-nickel alloys has been sufficiently studied [3] in terms of crystallography, thermodynamics of phase transformations and the mechanism of implementation, and current study is focused mainly on the practical use of such materials.

The operating conditions of products made of such alloys determine the requirements for these materials. Such requirements usually include the number of cycles without significant residual deformation, the temperature interval of the effect, mechanical properties, and others.

For the practical use of materials with the superelastic effect, it is necessary to obtain inseparable connections of elements from these materials, including by welding or soldering. The structure and phase composition of the weld material determine the properties of the connection. One of the most effective types of nitinol welding is laser [4]. Thus, the study of the structure of the weld joint of nitinol wires made by laser welding is promising for solving the current problem of finding ways to manufacture nitinol structures.

2. Literature review and problem statement

Thematic focus on welding of shape memory alloys, in particular nitinol, is observed in many studies [4–7].

The results of work [4] show that the thermal effect of laser radiation and recrystallization of the material in the welding zone preserve the possibility of shape recovery in the weld material. At the same time, the characteristic temperatures of the direct and reverse martensitic transformation increase. According to the Ti–Ni phase diagram [3] at a temperature of 800°C, the homogeneity interval of the Ti–Ni intermetallic compound is within 49–53 at.%, and at a temperature of 400°C it narrows to 49.5–51 at.% Ni. Outside the homogeneity region, the alloy consists of a mixture of $TiNi + Ti_2Ni$ phases with an excess of titanium and a mixture of $TiNi + TiNi_3$ phases with an excess of nickel. According to the results of [4], the possibility of thermally stimulated recovery of the shape of a laser welded joint made of nitinol is shown, but the choice of optimal technological modes is postponed to further studies.

As a result of a comprehensive review of studies of the TiNi alloy enriched with titanium, which was welded by laser [5], the negative effect of Ti_2Ni precipitation on the mechanical properties of both the heat-affected zone and the fusion zone is shown. The effect of laser welding on other functional properties of nitinol is also analyzed. Problems are shown regarding the reduction of the mechanical properties of the material in the welding zone. By solving the identified problems, it is proposed to search for effective modes of post-welding heat treatment. Carrying out such treatment also leads to changes in the beginning and end of phase transitions, which determine the return of the shape. The problem of preserving this effect within the limit's characteristic of the welded material while simultaneously ensuring the required level of joint strength remains unresolved.

Microplasma welding of nitinol with steel [6] made it possible to show the decisive influence of the difference in the coefficients of thermal expansion of the welded materials on the formation of cracks in the weld. The variety of intermetallics formed in the weld also does not contribute to a high level of mechanical properties of the joints. No final solution was found.

Promising results of performing welded joints from nitinol were shown in work [7]. The prospective use of solid-state welding methods was analyzed. At the same time, the low manufacturability of these methods limits the prospects of their use.

During the study of nickel-enriched nitinol [3, 8–11], it is assumed that if the NiTi alloy contains an excess of Ni, Ni_4Ti_3, Ni_3Ti_2, Ni_3Ti precipitates may form. In particular, work [8] shows a predominantly positive effect of Ni_4Ti_3 precipitates on the mechanical properties of the welded joint. In contrast, in Ti-enriched nitinol, only Ti_2Ni precipitates may be present. The features of intermetallic precipitates in TiNi alloys were carefully analyzed by the authors [10]. In [9], it was established that the formation of phases of this type can occur during the thermal cycle of the shape memory effect. In this case, precipitates of the type Ni4Ti3, Ni3Ti2, Ni3Ti significantly affect the course of martensitic transformations in nitinol [11]. The influence of the alloy composition on the transformation temperature varies significantly if it is Ni- or Ti-enriched nitinol. For Ni-enriched nitinol, the transformation temperatures decrease significantly with increasing Ni content. At the same time, increasing the Ti content from equiatomic does not lead to a significant change in the phase transition temperatures. Thus, the published results show that the influence of the phase composition formed in the nitinol weld is a determining factor that determines the operational properties of the joint. At the same time, it has not been possible to determine the composition and morphology of the phases in nitinol that would provide the maximum level of such properties. The reason for this may be objective difficulties associated with the ambiguous influence of intermetallics. A positive effect on mechanical properties may be accompanied by a negative effect on the temperature interval of phase transformations and vice versa. An effective way is to solve specific technological problems for individual products, but it is advisable to expand the spectrum of structural and phase transformations in the material of the welded joint.

Post-weld heat treatment is usually used to: remove residual stresses caused by the thermal cycle of welding [12] or optimize the mechanical properties of the welded joint material by obtaining a more suitable microstructure [13, 14]. Accordingly, the selection of recommended parameters of the heat treatment mode (temperature, time, option of thermal energy input) allows improving the mechanical properties of welded joints. The authors [5, 12–14] did not find a final solution, but a set of directions for searching for such solutions was formed.

Solutions for joint of metal elements from alloys for which the shape memory effect is inherent should provide for the preservation of the ability of unloaded materials under the action of external stress to accumulate up to 10–15% of the deformation energy. In the case of superelasticity, it is the deformation energy that returns to the previous shape in the process of removing the external load. To achieve a high-quality welded joint of nitinol, the authors of the study [4] propose to use a laser radiation power of 200–400 W at a welding speed of 500–5000 mm/min.

Thus, the main problems that exist when welding nitinol are changes in the elemental composition, phase composition and microstructure in the weld area and the heat-affected zone. This leads to changes in the parameters of phase transitions that ensure the implementation of the shape memory effect (including superelasticity), and, accordingly, to different implementation of this effect in materials in the weld area and the base material. This negative impact on the properties of the welded joint is often accompanied by the formation of brittle intermetallics near which the existing deformation stresses are able to ensure the existence of thermoelastic martensite, which limits the completeness of shape recovery.

All this allows to state that research devoted to determining the structural features of the material of the connection of nitinol wires can provide an additional tool for achieving the optimal structure of the material of the weld. New options for changing the laser welding cycles are needed. This is the way to determine the characteristics of the welding cycle, which will ensure the formation of a welded joint with an optimized structure of the seam that will meet operational requirements.

3. The aim and objectives of the study

The aim of the study is to ensure the formation of a thin nitinol wire joint by determining the structural and phase changes in the material of the weld, which are caused by the repetition of the laser welding cycle of nitinol wires. This will make it possible to regulate the structure of the material of the weld and ensure a state that preserves the properties of the material with a shape memory effect without thermal stimulation.

To achieve this aim, the following objectives were accomplished:

– to determine the structural characteristics of the joint material made by one and two cycles of laser welding of nitinol wires at a constant supplied thermal energy;

– to determine changes in the phase composition of the weld material, the connection of nitinol wires made by a repeated cycle of laser welding.

4. Materials and methods

4. 1. Characteristics of the joined materials, methods for obtaining permanent joints and preparation of samples for research

The object of the study is the welded joint of thin wires from the nitinol alloy. The main hypothesis of the study is the possibility of achieving new structural or phase components in the material of the weld by performing a repeated laser welding cycle.

The following assumptions and simplifications were made in the research: when choosing the parameters of the laser exposure modes on nitinol wires, it is assumed that the percentage of energy absorbed by the material of the joint does not depend on the duration of heating and radiation energy. Within the framework of this assumption, it is considered that the material absorbs the same energy when welding in modes 1 and 2.

To conduct research on the material of permanent joints of thin wires from the nitinol alloy, wires with a diameter of 0.8 mm were used. Permanent connections of nitinol alloy wires with a diameter of 0.8 mm were formed using a highly concentrated welding heat source – laser welding. This ensured high locality of thermal energy input, which contributes to minimizing the volume of the heating area, the size of the cast area of the welds and reducing the amount of residual stresses and deformations of the parts.

The chemical composition of nitinol alloy wires is given in Table 1.

Laser welding was performed using specially selected argon (Ar > 99.998% vol.) manufactured by Linde as a shielding gas. The content of impurity gases, determined by the results of the analysis of the argon composition, according to the gas manufacturer's certificate for the cylinder No. 13458, dated 05/10/2018, did not exceed: O – 0.00019% vol.; N – 0.0002% vol.; water vapor –0.0002% vol.

Table 1

Chemical composition of nitinol alloy wires

Material	Chemical composition, wt. %.								
	Ni	C	Co	Cr	Fe	Nb	Ti	Cl + Br	Other
Wire with a diameter of 0.1 mm to 3.0 mm according to ASTM F2063 [15]	54.5–57.0	≤ 0.05	≤ 0.05	0.15–0.3	≤ 0.05	≤ 0.025	Balance	≤ 0.15	O ≤ 0.05; H ≤ 0.005; N ≤ 0.009
Wire according to the certificate No. 20240711001, dated 11.07.2024, submitted by the wire manufacturer	55.65	≤ 0.05	≤ 0.01	0.25	≤ 0.01	≤ 0.005	Balance	≤ 0.01	O ≤ 0.05; H ≤ 0.002; N ≤ 0.003

To obtain non-separable connections of thin wires made of nitinol alloy with each other for experimental studies, elements were manufactured by mechanical cutting with a length of 35 mm. After mechanical processing, the elements were washed with acetone and treated with ethyl alcohol to remove possible contamination residues.

4. 2. Welding mode parameters, equipment and devices

To perform welded permanent joints, a laser welding installation manufactured by SENFENG (PRC) model SF 1500 HWM (Fig. 1) was used. The installation includes a RAYCUS RFL C 1500 laser radiation source (Fig. 2) with a power of 9 kW and a torch for manual welding (Fig. 3).

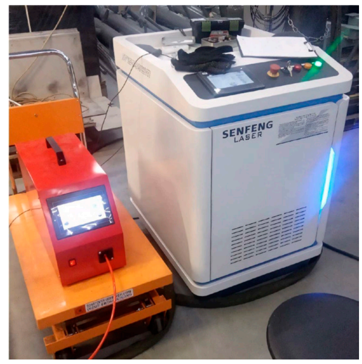


Fig. 1. General view of the laser welding system manufactured by SENFENG (PRC) model SF 1500 HWM

Fig. 2. General view of the laser radiation source RAYCUS RFL C 1500

Laser radiation from the RAYCUS RFL C 1500 yttrium source with a wavelength of 1080 nm was supplied via an optical fiber to the welding head. The welding laser head is equipped with a nozzle for supplying shielding gas to the heating area. The necessary pressure and flow rate of the shielding inert gas were set using regulators. Laser welding of the samples was performed without the use of filler wire.

The applied laser welding mode parameters are given in Table 2.

Immediately before jointing, the sections of the elements to be welded were mechanically processed using P1000 grade sandpaper. After fine grinding, the samples were washed with ethyl alcohol and installed in a device for fixing and compressing the elements during the welding process (Fig. 4). The device allows to obtain butt joints of elements from thin wire or thin tubes, adjust the protrusion of the samples from 1 mm to 40 mm and the amount of settlement from 0 mm to 5 mm, and adjust the effective compression force from 0.5 N to 10 N.

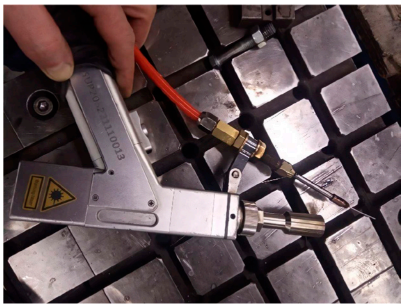


Fig. 3. Welding head for manual laser welding

Table 2

Recommended laser welding mode parameters (LW)

Welding method	Welding mode number	Laser power, W	Pulse frequency, kHz	Heating area diameter, mm	Heating time, s	Shielding gas flow (Ar), l/min
Laser welding (LW)	1	400	5	0.8	0.3	10
	2	600	5	0.8	0.1 twice	10

Before welding, elements made of thin wires were installed in a device for fixing and compressing elements (Fig. 4) and fixed. For wires with a diameter of 0.8 mm, the protrusion was 3 mm. Before the welding process, a protective inert gas of argon was supplied. After the welding process was completed, the supply of shielding gas was carried out for 20 s in order to protect the heated metal of the welded joint from the influence of the external atmosphere during cooling.

Fig. 4. Device for fixing and compressing elements during the welding process

4. 3. Study of the structure of the material of welded joints

To determine the structure and phase composition of the weld material, metallographic and micro-X-ray spectral analyses of welded nitinol wire samples were performed. For this purpose, they were pressed into bakelite in a steel cylindrical mandrel. The samples were mechanically ground until their diametrical cross-section was achieved. Subsequently, the samples were mechanically polished to a roughness of Rz ~ 0.2 μm.

Chemical etching of the studied samples to reveal the microstructure was carried out in a 49% solution of nitric acid (HNO_3) with the addition of 18% HF and 23% water, followed by washing with ethyl alcohol.

The samples were studied using a TESCAN VEGA 3 scanning electron microscope with an energy-dispersive X-ray analyzer (Brno, Czech Republic). Acceleration voltage range: 0–30 kV. Available detectors: SE, BSE, EDS. Resolution: 3–3.5 nm in SE mode.

The study was conducted in the secondary (SE) and backscattered electron (BSE) modes.

Secondary electrons are generated in thin near-surface layers of the material (1–5 nm), so they are sensitive to the surface state and provide information about the surface relief. Backscattered electrons are reflected from the sample surface due to elastic scattering (depth 1–5 μm), the signal intensity from them is directly related to the average atomic number of elements in the studied area. This allows to directly isolate areas of different composition. The electron probe current was 10^{-10}–10^{-8} A. This allowed to study the nature of changes in the features of individual structural fragments and elements.

For a detailed determination of the composition of elements in the test sample, the energy dispersive analysis (EDS) technique was used, which allows for microanalysis of dispersed particles and phases. The minimum size of the analyzed area depends on the composition of the material and is about 1 μm. The detection capability of most elements is in the range of 0.1–0.5 mass.%. For local analysis of elements with low concentrations, a second wavelength-dispersive spectrometer is used.

5. Results of the study of the structure and phase composition of the weld material made by laser welding

5. 1. Structural characteristics of the material of the joint made by laser welding

As a result of laser welding of nitinol wires welded in mode 1, no macrodefects were recorded in the material of the joint. Fig. 5 shows the structure of the material of the weld of nitinol wires formed in mode 1.

Analysis of the structure of the material of the obtained connection (Fig. 5) allows to determine the width of the weld seam in the middle zone of ~ 200 μm (wedge-shaped seam with a width on the surface of 800 μm). A feature of the structure of such a material in comparison with the base of the welded materials (light shade) is an increased concentration of dark phase inclusions. Such inclusions can be divided into two types (Fig. 5): accumulation of small-sized dark phase inclusions and large individual dark phase inclusions.

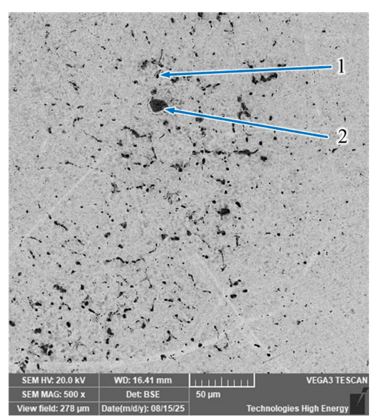


Fig. 5. Material structure of the weld seam made by laser radiation in mode 1: 1 – large individual inclusions of the dark phase; 2 – accumulation of small-sized dark phase inclusions

The structure of the material of the weld seam made by laser radiation in mode 1 is close to the structure of nitinol in a wire with a diameter of 0.8 mm (Fig. 6). The concentration of point inclusions in the weld material (mode 1) increases compared to the material in the nitinol wire.

As a result of laser welding of nitinol wires welded according to mode 2, no macrodefects were detected in the material of the joint. The structure of the material of the weld of nitinol wires formed according to mode 2 is shown in Fig. 7.

The main elements of such a structure are the light gray matrix of the base of the weld seam with two types of phases. These are dark gray phase formations with blurred interphase boundaries and eutectic-type zones formed by light gray and black phases of the lamellar type (typical of eutectics) (Fig. 8). The number of point phase inclusions in the material in the surface and central part of the weld seam is significantly reduced.

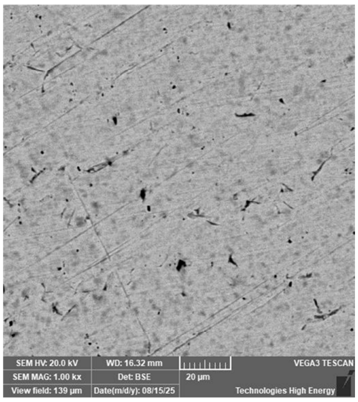


Fig. 6. Nitinol structure in a wire with a diameter of 0.8 mm

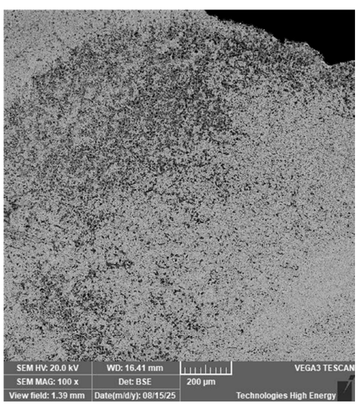


Fig. 7. Material structure in the surface and central part of the weld seam made by laser radiation in mode 2

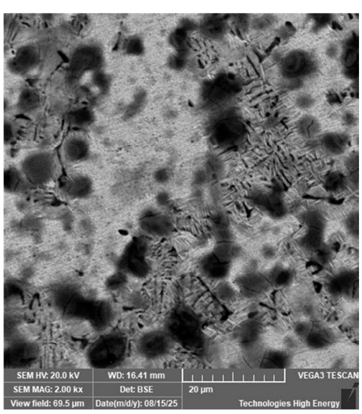


Fig. 8. Microstructure of the material in the near-surface and central part of the weld seam made by laser radiation in mode 2

The structure of the weld seam material in the root zone (Fig. 9) is close to the structure of the welded wires in the initial state (Fig. 7).

Thus, as a result of laser welding of nitinol wires with a diameter of 0.8 mm both by mode 1 and mode 2, a joint without macrodefects is formed.

For the materials of the weld in both welding modes, the presence of point formations in their structure is characteristic. In the structure of the materials of the weld formed by mode 2, grains are observed that have the appearance inherent in eutectic formations.

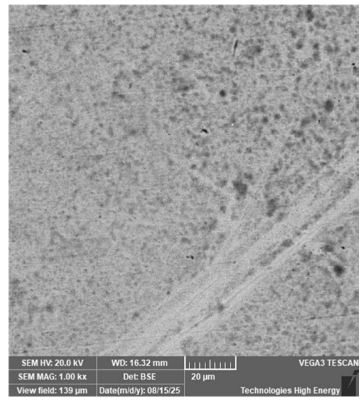


Fig. 9. Nitinol structure in the root region of the weld (by mode 2)

5. 2. Determination of changes in the phase composition of the material of the joint of nitinol wires, performed by a repeated welding cycle

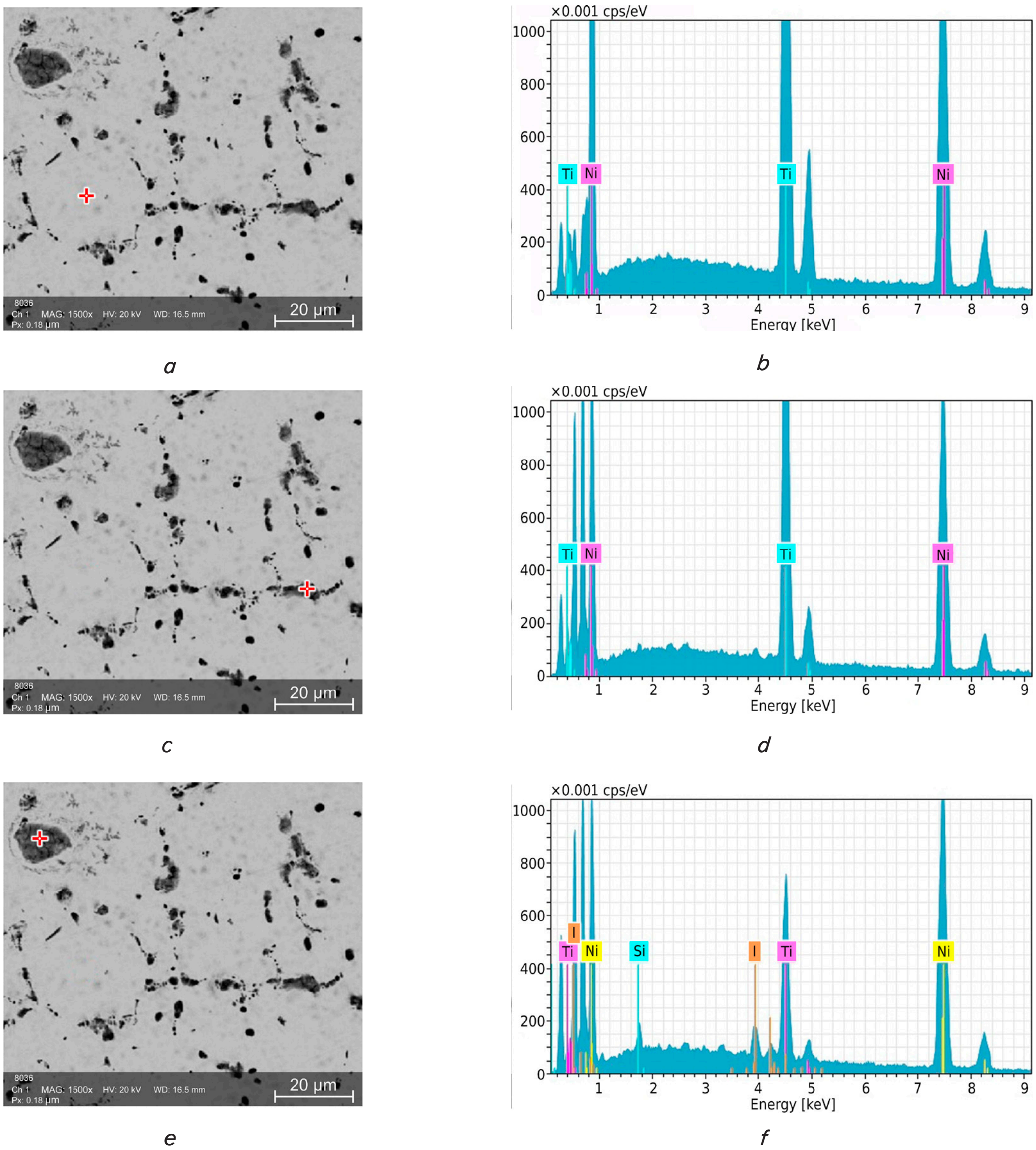
In the conditions of supply, the state of superelasticity is characteristic of nitinol wire. X-ray phase analysis of the welded wires showed that their material is in the state of TiNi austenite.

It was not possible to determine the phase state of the weld material by performing X-ray phase analysis due to the miniature size of the object of study. The probable phase composition was determined taking into account the concentration of elements detected by local chemical analysis.

The results of the elemental composition of the main structural components of the weld material obtained by mode 1 are shown in Fig. 10. It should be noted that the base material of the weld meets the ASTM F2063 standard within the margin of error, but is depleted in nickel content according to the manufacturer's certificate (Table 1).

The lack of nickel in the base material of the weld is compensated by its increased content in the composition of the dark phase inclusions, in which its concentration is recorded within 58.94–79.97 at.% Ni. At the same time, for nitinol welding wires in the initial state (Fig. 6) the content of 49.3–49.5 at.% Ni is characteristic in the presence of point phase formations of the type Ti_2Ni.

The obtained results show that the base material of the weld, formed according to mode 2, is close to the equiatomic composition in terms of titanium and nickel content and does not change in its form (Fig. 11, *a*) and elemental composition (Fig. 11, *g*) in comparison with the material of the welded wires (Fig. 6). Fluctuations in the nickel concentration in the areas of the main (light) phase do not exceed 0.6 at.%. The dark gray phase (Fig. 11, *c*) is close in composition to the weld base material with a slight increase in nickel concentration (Fig. 11, *h*). A new structural component is the eutectic grains (Fig. 11, *e*) with plates of the black phase containing up to 66 at.% Ni (Fig. 11, *i*). Such formations are stably observed until reaching the root zone of the weld with a gradual decrease in their concentration with depth.



Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	43952	43.97	47.86	52.95	1.27	2.89
Ni	28	22907	47.90	52.14	47.05	1.36	2.84
		Sum	91.87	100.00	100.0	–	–

g

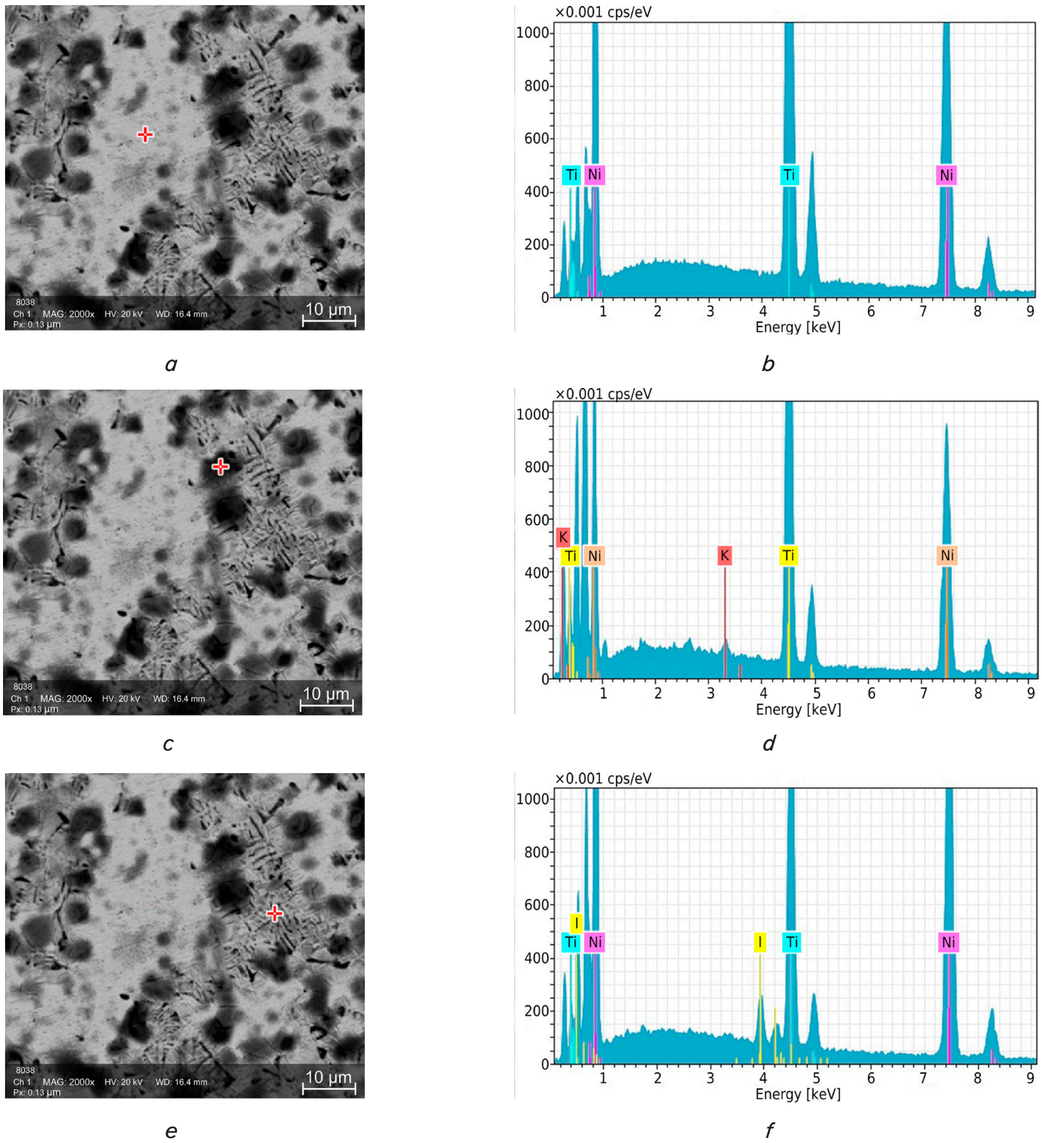
Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	20177	25.99	36.22	41.04	0.78	3.01
Ni	28	15651	45.78	63.787	58.96	1.33	2.91
		Sum	71.77	100.00	100.0	–	–

h

Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Si	14	710	0.74	1.11	2.26	0.09	11.92
Ti	22	20177	25.99	36.22	41.04	0.78	3.01
Ni	28	15651	45.78	63.787	58.96	1.33	2.91
I	53	1986	3.19	4.78	2.16	0.19	5.99

i

Fig. 10. Microstructure and results of analysis of the elemental composition of the main structural components of the material of the weld of nitinol (obtained by mode 1): *a* — section of the base material of the weld (marked with a cross); *b* — micro-X-ray spectral (EDS) characteristic of the base material of the weld; *c* — phase formation from the cluster in the composition of the weld material (marked with a cross); *d* — micro-X-ray spectral (EDS) characteristic of the phase formation from the cluster; *e* — coarse phase formation in the composition of the weld material (marked with a cross); *f* — micro-X-ray spectral (EDS) characteristic of the coarse phase formation; *g* — elemental composition of the base material of the weld; *h* — elemental composition of the phase formation from the cluster; *i* — elemental composition of the material of the coarse phase formation



Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	39018	38.77	45.093	50.17	1.13	2.91
Ni	28	21460	47.21	54.91	49.83	1.35	2.85
		Sum	85.98	100.00	100.00	–	–

g

Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
K	19	543	0.40	0.58	0.80	0.06	16.06
Ti	22	24260	29.19	42.79	47.69	0.87	2.98
Ni	28	13244	38.65	56.64	51.51	1.14	2.95

h

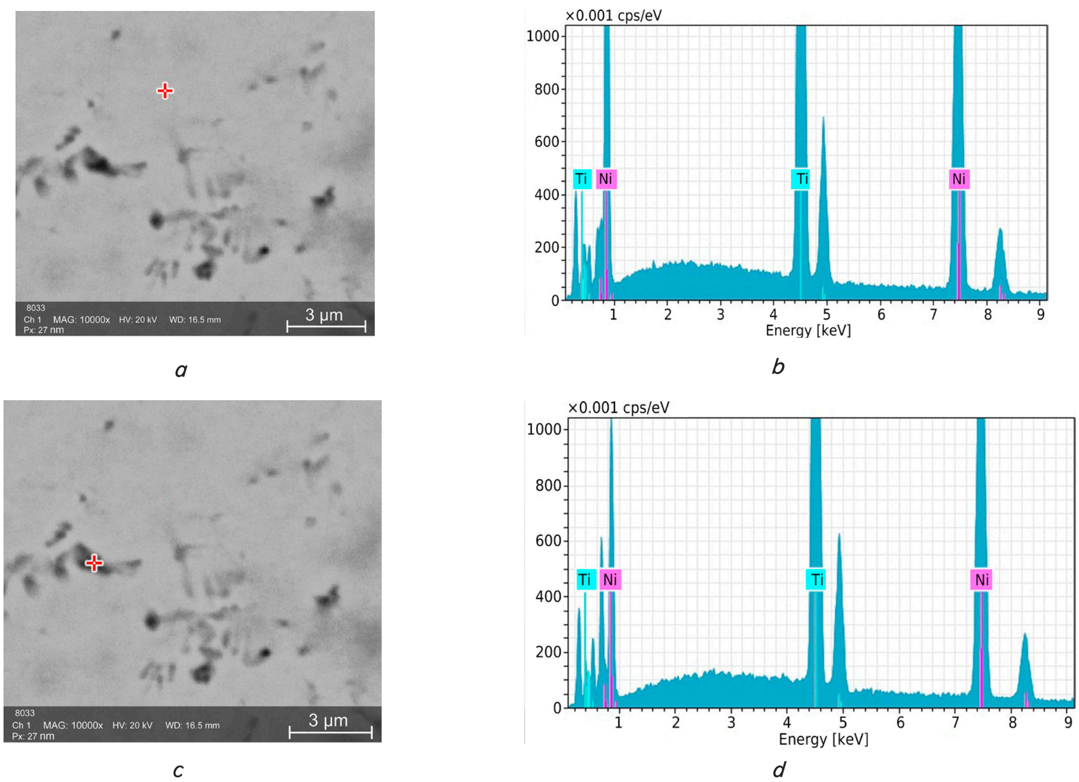
Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	17936	21.75	26.67	31.69	0.66	3.06
Ni	28	29655	55.48	68.04	65.94	1.58	2.85
I	53	3090	4.31	5.29	2.37	0.22	5.02
		Sum	81.54	100.00	100.00	–	–

i

Fig. 11. Microstructure and results of analysis of the elemental composition of the main phase components of the nitinol weld material (obtained by mode 2): *a* – section of the weld base material (marked with a cross); *b* – micro-X-ray spectral (EDS) characteristic of the weld base material; *c* – dark-gray phase formation in the weld material (marked with a cross); *d* – micro-X-ray spectral (EDS) characteristic of the dark-gray phase formation; *e* – eutectic type region in the weld material (marked with a cross); *f* – micro-X-ray spectral (EDS) characteristic of the eutectic type region; *g* – elemental composition of the weld base material; *h* – elemental composition of the dark-gray phase formation; *i* – elemental composition of the material of the eutectic type region

Analysis of the elemental composition of the main phase components of the Nitinol weld material in the root region (according to mode 2) showed (Fig. 12) that in this region the elemental composition of the main structural components is practically equiatomic in the base material (Fig. 12, *a*, *e*).

Point inclusions in the base material are characterized by a small nickel deficiency (Fig. 12, *c*, *e*).



Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	48397	44.13	45.62	50.70	1.27	2.88
Ni	28	27380	52.59	54.38	49.30	1.48	2.81
		Sum	96.72	100.00	100.00	–	–

e

Element	At. No	Netto	Mass, %	Mass Norm, %	Atom, %	abs. error, % (1 sigma)	rel. error, % (1 sigma)
Ti	22	45838	42.71	46.12	51.20	1.23	2.89
Ni	28	10925	49.90	53.88	48.80	1.41	2.82
		Sum	92.61	100.00	100.00	–	–

f

Fig. 12. Microstructure and results of analysis of the elemental composition of the main phase components of the root zone material of the nitinol weld (obtained by mode 2): *a* — section of the weld base material (marked with a cross); *b* — micro-X-ray spectral (EDS) characteristic of the weld base material; *c* — point phase formation in the composition of the weld material (marked with a cross); *d* — micro-X-ray spectral (EDS) characteristic of the point phase formation; *e* — elemental composition of the weld base material; *f* — elemental composition of the material of the point phase formation

The weld material in the main composition does not change the nickel content compared to the welded materials. Phases with an increased nickel content were detected in the form of point inclusions (welding by mode 1) and in the composition of eutectic grains, the number of which decreases with the depth of the weld (welding by mode 2).

6. Discussion of the results of determining structural and phase changes in the weld material

In the weld formed under the influence of laser radiation in mode 1, the intermetallic TiNi (Fig. 10, *g*) characteristic of nitinol is recorded as the base material by the ratio of titanium and nickel by EDS analysis. The presence of the Ti2Ni phase was not detected. The preservation of superelasticity in the welded joint when it was bent at an angle of 30° (residual deformation ~ 3°) gives reason to believe that the base material of the weld retains a predominantly austenitic state.

The phase formations of a dark shade in the form of clusters in the composition of the weld material (Fig. 10, *c*) have a slight deviation from the equiatomic composition of TiNi (Fig. 10, *h*) towards an increase in the nickel content. According to the Ti – Ni phase diagram [3], taking into account the content of chemical elements, this phase can be defined as the TiNi + $TiNi_3$ eutectic. At the same time, the content of the TiNi3 phase in such grains is insignificant. In the matrix of the main TiNi phase, grains of the phase with a titanium to nickel ratio of 1:4 are observed (Fig. 10, *e*, *i*). This ratio of elements corresponds to the eutectic (solid solution of Ti in Ni) + $TiNi_3$.

In the weld formed by laser welding in mode 2, the base material, taking into account the ratio of titanium and nickel, is the TiNi intermetallic (Fig. 11, *g*), typical of nitinol. When bending the welded joint of wires at an angle of 30°, the shape is preserved with a slight residual deformation of ~ 4°. The preservation of superelasticity in the welded joint gives reason to believe that the base material of the weld retains a predominantly austenitic state.

The dark phase with indistinctly expressed interphase boundaries (Fig. 9, *c*) has a small positive balance with respect to nickel (Fig. 9, *h*) and may be depleted in the TiNi + $TiNi_3$ eutectic. Areas with a typical eutectic appearance (Fig. 9, *e*) with an identified composition (Fig. 9, *i*), corresponding to the $TiNi + TiNi_3$ eutectic, are a characteristic feature of the material close to the thermal action of laser radiation. The amount of this phase decreases with distance from the surface that has undergone thermal action. It is absent in the material of the root zone of the weld formed by laser welding in mode 2. Two phases are observed in this zone (Fig. 11, *a*, *c*), characterized by a composition close to equiatomic (Fig. 11, *e*, *f*). The appearance of the point phase (Fig. 11, *c*) suggests that this phase is martensitic, unlike the base phase, which is austenitic.

To achieve the goal of ensuring the formation of a thin nitinol wire joint, it is advisable to obtain a welded joint without defects and phase components that worsen the complex of properties of the welded material. Such components include intermetallics and other phase formations that are equilibrium, and accordingly cannot be eliminated by further heat treatment without melting. The negative impact of such phase formations can manifest itself through the fragility of the welded joint or the loss or reduction of the manifestation of the superelasticity effect. A certain number of phases of this type are found in the welded materials in the initial state, but they are not registered by X-ray phase analysis, and are fixed by metallographic analysis as dispersed point inclusions of small concentration and size.

The results of the study of the structural characteristics of the material of the joints made by laser welding showed that the use of the recommended modes allows to obtain welded joints without macrodefects.

A single laser heating with a duration of 0.3 with an energy of 120 J leads to the formation of a weld with a significant number of clusters of grains that have a point or elongated character. Such grains are unevenly distributed in the base material, which, based on the results of EDS analysis, is identified as TiNi (Fig. 6, *g*), typical of nitinol. The presence of the Ti_2Ni phase in the TiNi grains was not detected. Given the insignificant supersaturation of the base material with titanium, inclusions of the Ti_2Ni phase were not detected due to their small size. Grains of inclusions that have a point or elongated character are defined as the $TiNi + TiNi_3$ eutectic.

According to [16], phases with a predominant nickel content contribute to the formation of martensite in the weld material. The appearance of martensite in austenitic nitinol was observed as a result of Nd:YAG laser welding of sheets with a thickness of 1000 μm with the appearance of Ni4Ti3 precipitates [16].

The $TiNi_3$ phase contributes to the implementation of the thermally initiated shape memory effect, but not the superelasticity of the material [8, 9, 11, 16]. Therefore, its existence should be considered a negative consequence of the formation of a weld seam when welding superelastic nitinol wires. Metallographic studies did not reveal martensite in $TiNi + TiNi_3$ inclusions, which is probably due to the small sizes (Fig. 6, *a*) of such inclusions. Moreover, even if martensite is formed in the composition of such a eutectic, its negative effect will be limited to small volumes of $TiNi + TiNi_3$ inclusions.

Inclusions (solid solution) $+ TiNi_3$ can have a more negative effect on the properties of the welded joint, since the effect of the presence of grains of this type on the superelasticity of nitinol has not been studied. A positive factor is the insignificant concentration of grains of this type in the weld seam material. The effect of inclusions of this type on the properties of welded joints of nitinol still needs to be studied.

As a result of double laser exposure to obtain a welded joint (mode 2), the heating duration is two times of 0.1 s with an interval of 1 s. The total energy supplied to the welded joint by the laser beam does not change – 120 J. With such exposure, the first thermal cycle not only forms the weld, but also heats the welding zone. This mode helps to eliminate possible defects of the first heating cycle, ensures the formation of dispersed phases, the nuclei of which were formed, but did not develop during the first thermal cycle. The formed structure of the welded joint is significantly different from the structure that was formed in mode 2. The density of the newly formed phases increases significantly in the near-surface zone, but it decreases with the depth of the weld. The main phase of the welded material does not lose its equiatomic composition. The main new structural components of the weld material are grains of the eutectic $TiNi + TiNi_3$, with a significant number of plates of the $TiNi_3$ phase. The appearance of TiNi in the structure of the eutectic (Fig. 11, *e*) suggests that it is in the martensitic state.

Analysis of the distribution of nickel by phase components of the weld material shows that the eutectic phase $TiNi + TiNi_3$ in the area of its observation is the only phase with an excess of nickel. In the root zone of the weld (Fig. 9, 11), where the eutectic phase $TiNi + TiNi_3$ was not formed, no significant deviations from the equiatomic composition of the structural components were detected. Point inclusions of Ti_2Ni, characteristic of the material of nitinol wires, are also not observed. This probably indicates that the entire weld material was heated to the melting point of Ti2Ni. The formation of individual inclusions in the martensitic state (Fig. 11, *c*) may be associated with the occurrence of thermal stresses, at a level sufficient for the formation of thermoelastic martensite.

The results obtained confirm the assessment [4, 5] regarding the suitability of laser welding for nitinol products of small thickness. At the same time, the obtained results show that by changing the multiplicity of thermal cycles of laser heating, significant changes in the structure of the weld can be achieved. In particular, it is possible to form grains of the eutectic $TiNi + TiNi_3$, which under other conditions are observed only as point inclusions.

It should be noted that, despite the large number of studies devoted to nitinol welding, the welding mode with a repeated cycle has not been used so far. It is under this mode that the formation of large-sized eutectic grains $TiNi + TiNi_3$ was observed. The discovery of the possibility of forming grains of the eutectic $TiNi + TiNi_3$, which under other conditions are observed only as point inclusions or metastable intermetallics, is the main scientific achievement of this study. Considering the mechanism of action of reheating on the welded joint, it can be considered promising to study the influence of changes in the initial temperature of the connected nitinol elements on the structure and material properties of the weld obtained by laser welding.

As a result of the analysis of the composition of point inclusions in the structure of the weld material, no phases were recorded that corresponded in composition to the Ti_3Ni_4, Ti_3Ni_2 phases, which do not correspond to the Ti – Ni phase diagram, therefore most of the phase formations were determined as eutectic. At the same time, it can be assumed that metastable dispersed, in particular nanoscale phases exist after welding in mode 1 and in mode 2 after the first stage of 0.1 s. Moreover, the existence of such inclusions in the welded joint after laser exposure is confirmed by the results of other studies [3, 8–11]. The second stage of laser energy supply leads to the disintegration of these metastable dispersed phase formations and the formation of a thermally stable eutectic. The absence of eutectic grains in the root zone of the weld (mode 2), in which the thermal effect of pulse heating is smaller, can serve as confirmation of this hypothesis. It is likely that the absence of registration of the $TiNi_2$ phase in the weld material is associated with the formation of its nuclei only at the nanolevel. The assumption about the existence of Ti_3Ni_4, Ti_3Ni_2, $TiNi_2$ phases in TiNi grains can be explained by fluctuations in the nickel concentration of 46.75 to 48.62 at.%. The answers to this hypothesis can be provided by the results of transmission electron microscopy with electronography.

The results of this study offer an additional tool for influencing the structure and phase composition of the weld material in the form of the multiplicity of the laser welding cycle. It is shown that the specified factor is effective, but the conditions for the positive influence of such a factor on the operational properties of the welded joint are not determined. The practical application of the proposed solutions is possible only based on the results of determining the entire complex of changes in the properties of the weld material: temperature intervals of phase transformations, mechanical properties of the welded joint, hysteresis of the shape memory effect, etc.

The main drawback of this study is the unreadiness of its results for practical application. The conditions for the onset and further growth of eutectic grains are uncertain. It is advisable to perform not only mechanical tests of the joints, but also thermal analysis, studies using transmission electron microscopy with the use of electronography. All this is methodologically difficult given the size of the research object, but in further studies these problems may be solved in model experimental conditions.

7. Conclusions

1. Changing the laser welding modes of thin nitinol wires from one cycle lasting 0.3 s to two cycles lasting 0.1 s at a constant heating energy of 120 J leads to the enlargement of the eutectic formations of $TiNi + TiNi_3$ in the weld material.

2. Changing the laser welding modes of thin nitinol wires from one cycle lasting 0.3 s to two cycles lasting 0.1 s at a constant heating energy of 120 J reduces the depletion of the nitinol base with nickel and promotes the localization of dispersed phases with an increased nickel content in the grains of the $TiNi + TiNi_3$ eutectic.

Conflict of interest

The authors declare that they have no conflict of interest regarding this study, including financial, personal, authorship or co-authorship.

Financing

The study was conducted with funding from the National Research Foundation of Ukraine for the implementation of scientific research and development for 2025 No. 2023.04/0074 "Precision connection for the manufacture of elements of medical biocompatible implants" according to Agreement No. 58/0074 dated March 3, 2025.

Data availability

Data will be provided upon reasonable request.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the presented work.

Authors' contributions

Viktor Kvasnytskyi: Conceptualization, Supervision, Writing – review & editing, Project administration, Funding acquisition; **Anastasiia Zvorykina:** Methodology, Formal analysis, Investigation, Data Curation, Writing – original draft; **Leonid Zvorykin:** Methodology, Investigation, Writing – original draft, Writing – review & editing; **Constantine Zvorykin:** Validation, Data Curation, Visualization; **Valery Kostin:** Methodology, Resources, Investigation; **Tatjana Taranova:** Resources, Investigation, Data Curation.

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