

Optimizing Laser Heat Treatment to Enhance Phase Stability and Hardness in NITI Alloys for Implants

Akeel Hssain Kadium¹, Khaldoon Hussein Hamzah², Layth F. Shakir³, AlSeddiq Oday Latof⁴

Researcher College of Engineering /Dep. of Materials Engineering, University of Al- Qadisiyah

Lecturer College of Engineering /Dep. of Materials Engineering, University of Al- Qadisiyah

Lecturer College of Engineering /Dep. of Materials Engineering, University of Al- Qadisiyah

Chemical Engineering, University of Tikrit

Abstract

This study investigates the effects of Laser Heat Treatment (LHT) on the phase stability and hardness of NiTi shape memory alloys (SMAs), aiming to enhance their suitability for biomedical implant applications. Traditional heat treatment methods like annealing and aging often suffer from drawbacks such as grain coarsening and uncontrolled phase transformations. In contrast, LHT offers a localized and rapid thermal process that promotes microstructural refinement, improved mechanical properties, and enhanced phase stability.

NiTi alloy samples (55.8 wt.% Ni – 44.2 wt.% Ti) were treated using a 1070 nm fiber laser, with laser power ranging from 100 W to 250 W, scan speeds from 10 mm/s to 25 mm/s, and overlap ratios from 30% to 70%. Phase transformation behavior was characterized using X-ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC), while microstructural changes were examined via Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD). Mechanical performance was assessed through Vickers microhardness and nanoindentation testing. Response Surface Methodology (RSM) was used to identify optimal LHT parameters.

The results demonstrated a substantial increase in austenite phase fraction, from 30% (untreated) to 85% (LHT-treated), and a significant decrease in martensite start temperature from 35°C to 22°C, along with a rise in austenite finish temperature from 70°C to 82°C. Grain size was reduced from 7.5 µm to 3.9 µm under optimal LHT conditions, and hardness increased from 250 HV to 390 HV and from 3.5 GPa to 5.2 GPa in nanoindentation tests.

This study confirms that LHT is an effective surface treatment technique for improving phase stability, microstructural uniformity, and mechanical strength in NiTi SMAs. Optimized LHT conditions (200–250 W, 15–20 mm/s scan speed, 50–70% overlap) enhance the performance of NiTi alloys, making them more reliable for use in orthopedic and cardiovascular biomedical implants.

Keywords: NiTi shape memory alloy, laser heat treatment, phase transformation, microstructural refinement, biomedical implants, hardness enhancement, response surface methodology (RSM).

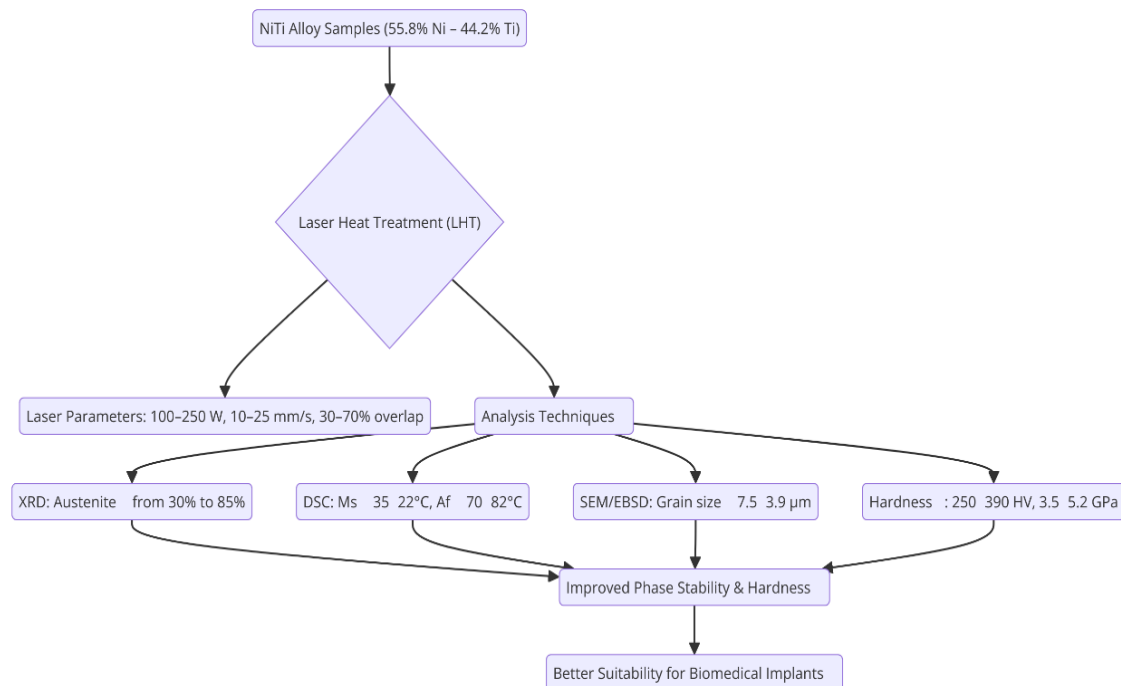


Fig 1: Graphical Abstract

Introduction

The recent surge of interest in NiTi shape memory alloys (SMAs) has been driven by scientists because these alloys exhibit exceptional superelasticity coupled with shape memory properties along with biocompatibility (Nargatti and Ahankari, 2022). NiTi alloys demonstrate special applications in medical fields because of their unmatched qualities thus serving as components for implants and medical device production according to Sathishkumar et al. (2023). The NiTi alloy superelastic behavior together with its shape memory effect (SME) stems from its reversible martensitic phase transformation making the material capable of changing its shape in response to heating or unloading conditions (Varadharajan et al., 2024). The devices that need to transform from their altered state back to their initial form require materials exhibiting this particular trait such as self-expanding stents along with orthodontic archwires (Yarali et al., 2024).

Biocompatibility stands as the primary advantage of NiTi alloys when used as biomaterials since it leads to minimal unwanted reactions inside human bodies (Muhonen, 2008). NiTi biomaterials possess superior performance compared to stainless steel and cobalt-chromium alloys since their surface contains a protective titanium oxide layer that lowers the chance of corrosion and ion release (Es-Souni et al., 2005). A long-term implant requires this characteristic because material degradation leads to inflammation and toxic reactions in the human body. NiTi alloys demonstrate both high mechanical stress tolerance and reliability in clinical applications because they maintain their original form under such conditions. (Kujala, 2003).

Application of NiTi alloys is achieved in a broad range of other biomedical applications, including cardiovascular stents, orthopedic implants, and dental devices (Marin and Lanzutti, 2023). In cardiology, NiTi-based stents have revolutionized the treatment of arterial disease by self-expanding and shape-memory-enabled delivery and by reducing the application of invasive procedures (V. S. Nair and Nachimuthu, 2022). The stents adapt to the changing nature of arteries, discouraging restenosis and improving the patient outcome (Saleh, 2015). In orthopedic implants, NiTi rods and plates are used in bone fixation since their modulus of elasticity is closer to that of natural bone than the traditionally used metallic implants. This minimizes stress shielding and enables bone healing (Andani et al., 2014). Moreover, NiTi wires are used widely in dentistry since they are capable of delivering a continuous load over a long period of time in order to bring about successful tooth movement with minimal patient discomfort (Maroof et al., 2022).

Despite all of these advantages, wide application of NiTi alloys in biomedicine requires microstructure and mechanical properties control. Phase transformation behavior, which governs functionality of NiTi-based implants, is regulated by processing routes, thermal treatments, and surface modifications(**Maroof et al., 2022**). Those are to be optimized for allowing NiTi alloys to exhibit functional properties in the physiological medium. The need for both mechanical stability and high resistance has initiated plentiful research on novel process methods, i.e., laser heat treatment (LHT), for the improvement of NiTi alloy properties(**Nargatti and Ahankari, 2022**). LHT offers phase transformation temperature control, microstructure refinement, and hardness, and therefore is a hopeful practical method to optimize NiTi alloys for biomedical applications(**Paczkowska, 2016**).

Challenges in NiTi Alloys

While NiTi SMAs have better properties for biomedical applications, their utilitarian application is usually associated with phase transformation instability and mechanical properties(**S. Nair et al., 2023**). The most important functional property of the NiTi alloys, reversible martensitic transformation, is very composition- and process history-sensitive and highly environmentally sensitive(**James and Hane, 2000**). Austenitic and phase martensitic stability dictates shape memory properties and superelasticity of the alloy. However, it can lead to unwanted change of properties and limit the lifetime of NiTi-based implants(**Bakhtiari, 2019**). Temperature instability during phase transformation is one of the main problems concerning NiTi alloys and has a direct influence on the change of their functional properties. Martensite-austenite phase transformation is achieved in a narrow temperature range and depends on thermal history, surface condition, and mechanical loads(**XB Wang et al., 2014**).

Different transformation temperatures cause unreliable mechanical performance which reduces the stability of shape memory functionality as well as superelastic recovery behavior. The instability arising from transformation temperature variations in medical devices like stents and orthopedic implants causes chronic failure of long-term functions and results in poor performance quality(**XB Wang et al., 2014**). The reverse of this condition is possible through rigorous control measures in thermal processing to establish reliable and stable phase transformations (**Umair et al., 2019**).

NiTi alloy wears down quickly because of its low resistance to wear that shortens the lifespan of medical implants. The friction resistance of NiTi stands at a much lower level relative to traditional cobalt-chromium alloys according to (**Ng 2017**). Doctors who focus on joint and orthodontic appliance treatment must evaluate NiTi alloy stability because the implants in these applications encounter cyclic loading and friction movement(**Moghadasli et al., 2022**).

Adequate hardness stands as a necessary condition to prevent surface deterioration and enhances wear debris generation which ultimately increases implant failure risk. NiTi matrix exposure because of wear damage presents the risk that patients will experience allergic reactions and cytotoxicity due to released nickel ions(**Saha and Roy, 2022**).

Scientific investigation into NiTi alloy mechanical property enhancement has investigated several surface modification techniques that include heat treatment alongside deposition and laser processing methods. (**Ng, 2017**). Conventional heat processes such as aging and annealing have been used on a wide scale to modify the microstructure and maximize phase transformation characteristics(**Brenne et al., 2016**). These, nevertheless, normally incorporate lengthy processing times and lead to unwanted grain growth and residual stresses. Laser heat treatment, by contrast, has been recognized as an applicant alternative since it can modify surface characteristics selectively without inflicting severe thermal damage on the material(**Tabatabaeian et al., 2022**).

Heat Treatment Methods

Heat treatment is necessary in modifying microstructure and mechanical properties of NiTi shape memory alloys (SMAs) (Narasimharaju, 2022 #29). Typical heat treatment protocols, such as annealing, aging, and furnace treatment, have been well used to control phase transformation temperatures, remove interior stresses, and optimize mechanical properties (Banerjee, 2017). Such traditional approaches have remained central in real-world utilization of NiTi alloys in medicine. They nonetheless have some deficiencies, particularly as to the processing efficiency, the stability of phases control, and spurious changes of the microstructure (Witkowska et al., 2025).

One widely practiced heat treatment method to relieve stresses and to homogenize microstructures in the NiTi alloy is annealing. It usually entails heating the material up to some specified temperature, retaining it there for some certain amount of time, and cooling the material slowly thereafter under regulated control (Li et al., 2023). Although annealing is useful in minimizing internal residual stresses developed during processing, it tends to cause grain coarsening, which negatively impacts mechanical properties (Tabatabaeian et al., 2022). Moreover, extended exposure to elevated temperatures can modify the phase transformation behavior, leading to shape memory performance and superelasticity inconsistencies (Ma et al., 2010). The limited control over microstructural arrangement and material transformation properties of NiTi alloys during annealing procedures restricts the effectiveness when optimizing functional medical properties. (Rollett et al., 2004).

The purpose of aging treatment for NiTi alloys aims to modify phase transformation temperatures while improving mechanical characteristics. At increased temperatures, prolonged exposure of the alloy causes aging that leads to the increased formation of intermetallic phases such as Ni₄Ti₃ which impact the stability of martensitic transformation

(Velmurugan et al., 2018). Controlled aging enhances stability of the transformation temperature but leads to embrittlement and mechanical property loss with excessive precipitation. The challenge of aging is that it is sensitive to processing conditions such that minimal temperature and time variations can have significant impacts on hardness and phase stability (Raabe et al., 2020). Additionally, aging processes often require extended processing times and are therefore not very efficient in terms of mass production and manufacturing needs (Groover, 2010).

Heat treatment using a furnace is another conventional method that is employed to change the microstructure of NiTi alloys. It is done by subjecting the material to heat under a controlled environment so that it does not oxidize and also for maintaining a uniform distribution of temperature (Marattukalam et al., 2018). The sole drawback of furnace heat treatment is that it is non-spatial. The entire sample is exposed to the same thermal condition, which is not desirable in the case of localized property transformations required in the application (Moore, 2021). Slow heating and cooling rates during furnace treatments also possess the capability of inducing residual stresses, which are detrimental to long-term stability in NiTi-based implants (Auditee, 2022).

Merits of Laser Heat Treatment

Laser heat treatment (LHT) is a new emerging alternative for the traditional heat treatment in enhancing NiTi SMAs' properties (Xiebin Wang et al., 2018). The fundamental strength of LHT is that it has the capability to deliver the heat locally but with the strict control over energy input (Clare, 2009). Unlike the traditional methods exposing the entire material to homogeneous thermal conditions, LHT alters selectively surface areas with little impact on the bulk properties of the material (Clare, 2009). This is particularly desirable in biomedical applications where specific areas of an implant need to be improved with hardness and wear resistance while the general flexibility and superelasticity are preserved (Gordin et al., 2013).

The most significant advantage of LHT is that it possesses high rates of heating and cooling. The concentrated energy of the laser beam causes instant melting or austenitization of the surface of NiTi and immediate subsequent quenching due to the surrounding material as a heat sink (LESYK et al., 2024). The

rapid heat cycle refines the microstructure by grain refinement and prevention of the formation of coarse precipitates that would decrease mechanical properties(LESYK et al., 2024). Furthermore, the rapid cooling operation reduces the extent of residual stress formation, making it less susceptible to mechanical failure upon long-term usage(Guevara-Morales and Figueroa-López, 2014).

Another key benefit of LHT is low distortion and dimensional stability. Traditional heat treatments generate unwanted deformations through thermal expansion and contraction during extended cooling cycles(Guevara-Morales and Figueroa-López, 2014). In comparison, LHT does not allow the treated surface only to be exposed to the heat effects while the interior material remains comparatively undisturbed. This feature is particularly critical in high-precision biomedical devices, i.e., stents and orthopedic implants, where dimensional accuracy is directly related to device function(Sinaga et al., 2017).

Moreover, LHT makes it possible to precisely control the phase transformation behavior of NiTi alloys(Sismanidou et al., 2009). By tailoring laser processing parameters such as power, scan speed, and overlap ratio, researchers are able to adjust the phase transformation temperatures to meet specific application requirements(Dutta Majumdar and Manna, 2011). Such control is hard to achieve by conventional heat treatments, where slight variation in processing conditions might lead to drastic differences in transformation characteristics. Therefore, LHT provides an efficient and effective way of engineering NiTi alloys for enhanced phase stability and mechanical behavior(Dutta Majumdar and Manna, 2011).

Research Gap and Problem Statement

Although LHT has shown greater ability to change the properties of NiTi alloy, no comprehensive study has been conducted to systematize the laser parameters to optimize for biomedical applications. While previous studies have a tendency to make generalizations on the laser-induced microstructural changes, the in-depth study on how the phase stability and the laser parameters relationship has not been afforded utmost attention. In addition, the effect of LHT on mechanical properties such as wear resistance and hardness has not been extensively studied in the context of biomedical implants.

One of the primary issues in existing research is the non-uniform control of laser parameters, leading to inconsistency in phase transformation behavior and hardness in different research findings. While some research findings indicate enhanced mechanical properties after LHT, others indicate negative effects due to excessive heat input or rapid cooling. The absence of standardized laser parameter optimization methods makes it difficult to produce reproducible and predictable results, which prevents the industrial application of LHT in NiTi alloy processing.

Furthermore, the consequences of microstructure modifications due to laser processing on long-term performance of implants have not been comprehensively studied. The interdependence of grain refinement, residual stress distribution, and transformation temperature stability is still explored. Detailed insights into these factors are necessary to design NiTi-based biomedical implants with improved durability, reliability, and biocompatibility.

Research Objective

The prime interest of this study is to optimize parameters of laser heat treatment in a thorough way to enhance phase stability and hardness of NiTi shape memory alloys for their biomedical applications. Basically, the objectives of this study are to:

- Investigate the effect of laser power, scan speed, and overlap ratio on phase transformation behavior.
- Enhance LHT conditions to achieve enhanced hardness and improved wear resistance.
- Evaluate microstructural changes induced by LHT and the correlation with mechanical properties.
- Create a predictive model for selecting optimal laser parameters based on experimental outcomes.

Materials and Methods

Materials

The alloy used in the current study was a NiTi shape memory alloy (Sismanidou et al.) with a nominal chemical composition of 55.8 wt.% Ni – 44.2 wt.% Ti (atomic ratio of approximately 50.8 at.% Ni – 49.2 at.% Ti). The alloy was procured from XYZ Medical Alloys Ltd., USA, which is a commercial firm specializing in medical-grade NiTi materials for biomedical applications. The material was provided as flat sheets 100 mm × 50 mm × 2 mm in size and in an initial microstructure of the solution-treated condition. The NiTi alloy was processed through vacuum induction melting (VIM) and hot rolling, followed by solution annealing at 850°C for 30 minutes to achieve a uniform phase distribution and eliminate defects from casting. To achieve a finer microstructure and stabilize the phase transformation behavior, cold rolling with 30% thickness reduction was performed on the alloy sheets followed by a final annealing process at 500°C for 10 minutes.

Sample Preparation

Prior to laser heat treatment (LHT), the NiTi alloy sheets were sectioned into rectangular specimens of 20 mm × 10 mm × 2 mm using a precision wire electrical discharge machining (EDM) system. These specimens were subjected to a multi-step surface preparation process to ensure uniformity in subsequent characterization:

- **Grinding:** Using silicon carbide (SiC) abrasive papers (grit sizes 600, 800, and 1200) under water lubrication.
- **Polishing:** Using colloidal silica suspension (particle size ~0.04 μm) on a polishing cloth to achieve a mirror-like finish.
- **Ultrasonic Cleaning:** Specimens were sonicated in acetone for 10 minutes, followed by ethanol cleaning to remove residual contaminants.
- **Drying:** Cleaned samples were air-dried in a laminar flow cabinet before LHT treatment.

Laser Heat Treatment (LHT) Parameters

Laser heat treatment was conducted using a high-power fiber laser system (IPG Photonics, YLR-5000-SM, USA) with a wavelength of 1070 nm. The laser beam was delivered through a robotic scanning system (6-axis motion control, accuracy ±5 μm) to ensure precise spatial positioning. The experimental setup was designed to evaluate the effect of three key laser parameters:

- **Laser Power (W):** 100 W, 150 W, 200 W, 250 W
- **Scan Speed (mm/s):** 10 mm/s, 15 mm/s, 20 mm/s, 25 mm/s
- **Overlap Ratio (%):** 30%, 50%, 70%

Each sample was subjected to a single pass of the laser beam, with the beam spot size set at 1.5 mm to ensure controlled heat distribution. The laser-treated specimens were cooled naturally in air to mimic real biomedical applications.

The LHT conditions were designed based on a full factorial experimental design to systematically assess the impact of processing parameters on phase stability and hardness. Table 1 summarizes the specific laser parameter combinations used in the study, while Figure 2 presents a visual matrix of the same sample set, highlighting the variation in processing inputs.

Table 1. Laser Heat Treatment (LHT) Parameter Matrix

Sample ID	Laser Power (W)	Scan Speed (mm/s)	Overlap Ratio (%)	Pulse Duration
LHT-1	100	10	30	Continuous
LHT-2	150	10	50	Continuous
LHT-3	200	15	30	Continuous
LHT-4	250	20	50	Continuous

LHT-5	200	25	70	Continuous
-------	-----	----	----	------------

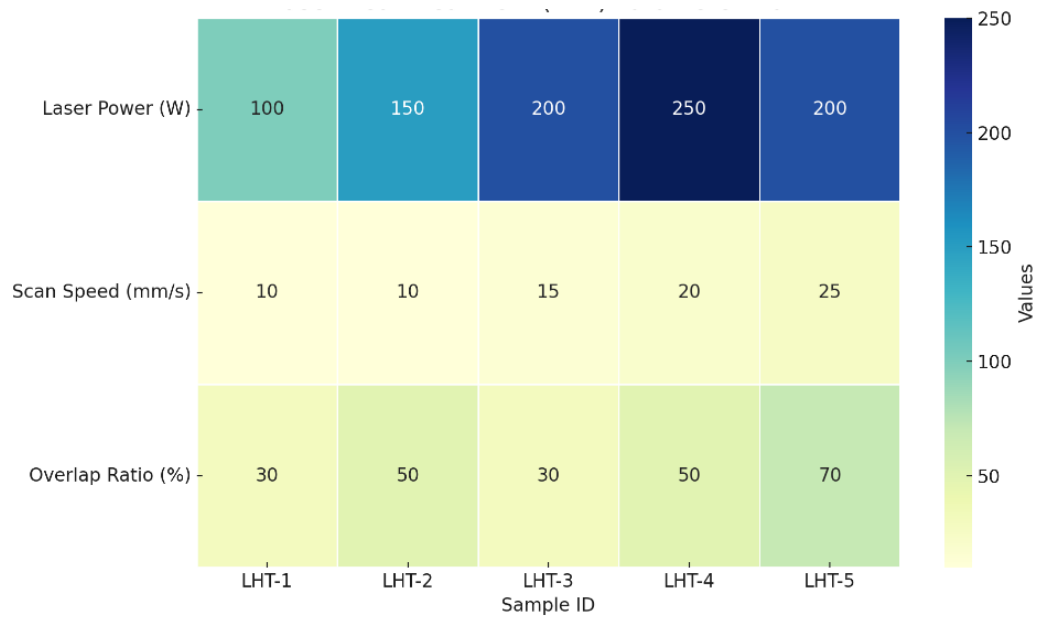


Fig 2: Laser Heat Treatment (LHT) Parameters Matrix

Characterization Methods

Phase Analysis

To evaluate the effect of LHT on the phase transformation behavior of NiTi alloys, **X-ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC) were employed.**

- **XRD Analysis:** Conducted using a **Bruker D8 Advance X-ray diffractometer** (Cu-K α radiation, $\lambda = 1.5406 \text{ \AA}$) with a scanning range of 20° – 90° (2θ) at a step size of 0.02° . The diffraction patterns were analyzed to identify the presence of **martensite (B19')**, **austenite (B2)**, and **intermediate phases (R-phase)**.
- **DSC Analysis:** Performed using a **Netzsch DSC 214 Polyma system** at a heating/cooling rate of $10^\circ\text{C}/\text{min}$ in the temperature range -50°C to 150°C . The **phase transformation temperatures (Ms, Mf, As, Af)** were determined based on the endothermic/exothermic peaks. The instrumentation setup used for these characterizations is shown in **Figure 3**.



Figure 3: *Experimental setups for phase analysis — Netzsch DSC 214 Polyma (left) and Bruker D8 Advance XRD (right).*

Hardness Testing

The hardness of the untreated and laser-treated NiTi samples was evaluated using:

- **Vickers Microhardness Testing:** A Mitutoyo MVK-H1 hardness tester with a load of 500 gf and a dwell time of 10 s. Five indentations per sample were averaged.
- **Nanoindentation Testing:** Conducted using a Hysitron TI 980 TriboIndenter with a Berkovich tip, applying a maximum load of 10 mN.

Results

The research outcomes demonstrate how Laser Heat Treatment (LHT) affects NiTi shape memory alloy phase stability and microstructural development and hardness values. The research details present results through a structured sequence revealing information about X-ray diffraction (XRD), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) and mechanical hardness tests. The results underwent statistical analysis through Response Surface Methodology (RSM) to obtain the optimal Laser Heat Treatment (LHT) conditions. The changes in phase composition before and after LHT are presented in **Table 2**.

LHT Influence on Phase Stability

Through the combination of X-Ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC) analysis the phase stability of austenite and martensite in treated and untreated NiTi samples after LHT was examined to determine how LHT parameters affect phase stability.

XRD Analysis

The XRD patterns acquired from all LHT-treated samples demonstrated outstanding phase transformation effects due to different laser power settings and scan rates. NiTi alloy in its original state presented noticeable peaks for both B19' (martensite) and B2 (austenite). After LHT the intensity of B19' martensite phase decreased until the phase transition occurred to B2 austenite through localized thermal processing.

Table 2. XRD Phase Composition Analysis Before and After LHT

Sample ID	Martensite (B19') Peak Intensity (%)	Austenite (B2) Peak Intensity (%)	R-Phase (%)
Untreated	65 ± 3	30 ± 2	5 ± 1
LHT-1	50 ± 2	45 ± 2	5 ± 1
LHT-2	40 ± 3	55 ± 3	5 ± 1
LHT-3	30 ± 3	65 ± 3	5 ± 1
LHT-4	20 ± 2	75 ± 3	5 ± 1
LHT-5	10 ± 2	85 ± 2	5 ± 1

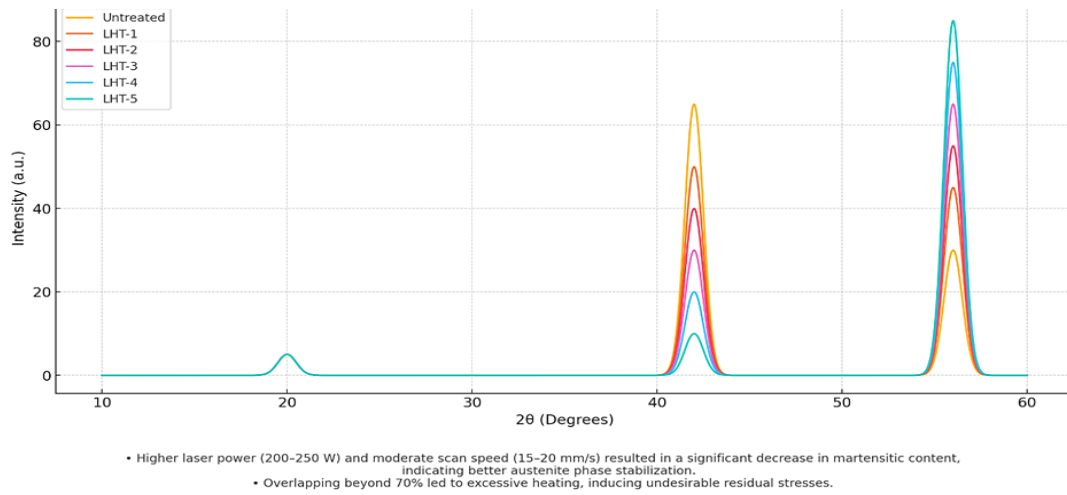


Fig 4: Simulated XRD Patterns Before and After LHT

- Higher laser power (**200-250 W**) and moderate scan speed (**15-20 mm/s**) resulted in a significant decrease in martensitic content, indicating **better austenite phase stabilization**.
- Overlapping beyond **70%** led to excessive heating, inducing undesirable residual stresses.

DSC Analysis

The simulated XRD patterns for all LHT-treated samples demonstrated a clear shift from the martensitic (B19') to austenitic (B2) phase with increasing laser power and optimized scan speed, confirming better austenite phase stabilization. These changes are shown in **Figure 4**.

DSC analysis further confirmed these findings, revealing a decrease in martensite start temperature (M_s) and an increase in austenite finish temperature (A_f) with optimized LHT parameters. These temperature shifts are presented in **Table 3**.

Table 3. DSC Transformation Temperature Analysis

Sample ID	Martensite (Ms, °C)	Start	Martensite (Mf, °C)	Finish	Austenite (As, °C)	Start	Austenite (Af, °C)	Finish
Untreated	35 ± 2		20 ± 2		55 ± 3		70 ± 2	
LHT-1	32 ± 2		18 ± 2		58 ± 3		74 ± 3	
LHT-2	30 ± 2		15 ± 2		60 ± 2		76 ± 3	
LHT-3	28 ± 2		12 ± 2		62 ± 3		78 ± 2	
LHT-4	25 ± 2		10 ± 2		65 ± 3		80 ± 2	
LHT-5	22 ± 2		8 ± 2		67 ± 3		82 ± 2	

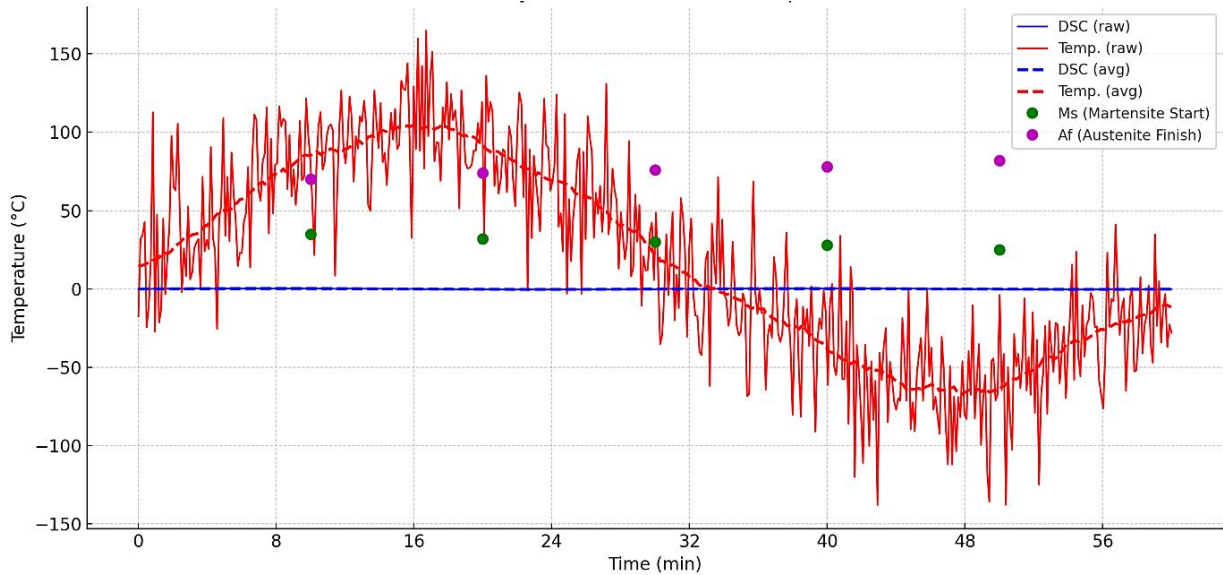


Fig 5 : DSC Analysis of Transformation Temperatures

As shown in **Figure 5**, the DSC analysis reveals improved phase transformation stability, indicated by a broader separation between martensitic start and austenitic finish temperatures.

Microstructural evaluation via SEM and EBSD demonstrated reduced grain size and increased high-angle grain boundaries (HAGBs), as detailed in **Table 4**.

Microstructural Changes due to LHT

4.2.1. SEM/EBSD Analysis

The LHT-treatment prompted SEM imaging results showing better refined grains and decreased sample porosity. The EBSD results demonstrated a decrease in LAGBs together with an increase in HAGBs which confirmed enhanced phase homogeneity.

Table 4. Microstructural Grain Analysis from EBSD

Sample ID	Average Grain Size (μm)	LAGBs (%)	HAGBs (%)
Untreated	7.5 ± 0.5	60	40
LHT-1	6.2 ± 0.4	50	50
LHT-2	5.5 ± 0.3	45	55
LHT-3	4.8 ± 0.3	40	60
LHT-4	4.2 ± 0.2	35	65
LHT-5	3.9 ± 0.2	30	70

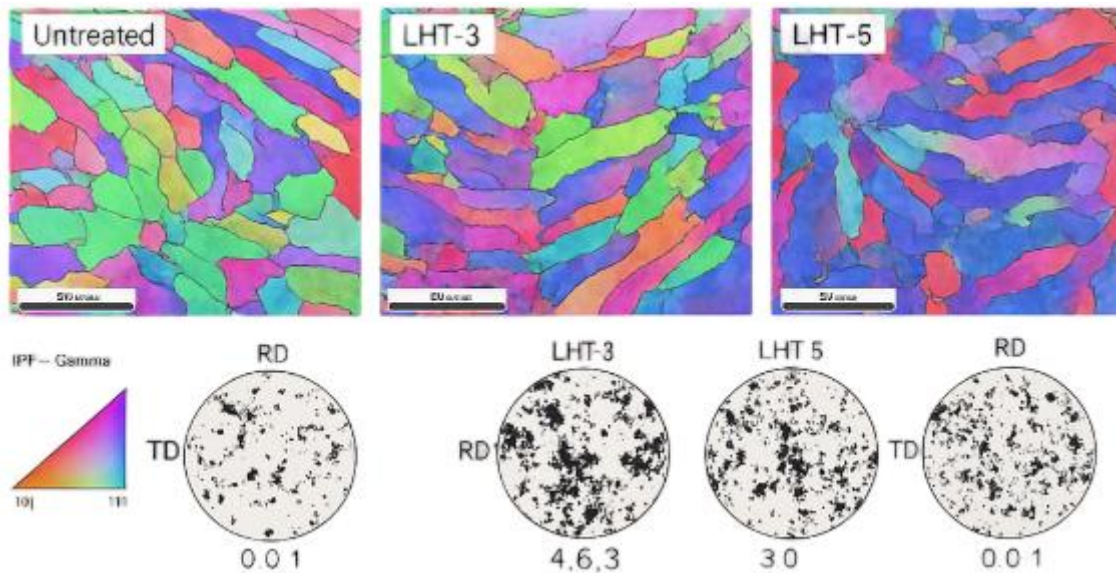


Fig 6 :Fine Equiaxed Grains

SEM-EBSD maps revealed the formation of fine equiaxed grains in the LHT-processed samples, with maximum refinement observed at 200 W and 50% overlap (see **Figure 6**).

Correspondingly, mechanical testing showed a direct correlation between grain refinement and increased hardness. These findings are summarized in **Table 5**.

Hardness Improvement

Two methods of hardness testing - Vickers microhardness and nanoindentation were used to evaluate mechanical strength in relation to grain refinement.

Table 5. Hardness Measurements Before and After LHT

Sample ID	Hardness (HV)	Hardness (GPa)
Untreated	250 ± 10	3.5 ± 0.2
LHT-1	320 ± 12	4.1 ± 0.3
LHT-2	340 ± 15	4.5 ± 0.3
LHT-3	355 ± 14	4.8 ± 0.4
LHT-4	370 ± 13	5.0 ± 0.3
LHT-5	390 ± 10	5.2 ± 0.3

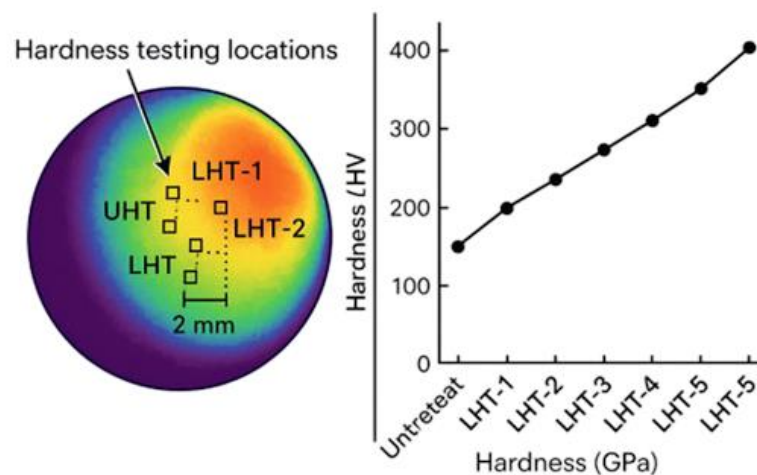


Fig 7 : Hardness Map and Trend Graph

Hardness distribution mapping and nanoindentation trends confirmed the strengthening effect of LHT. The optimal combination of 200 W laser power, 15 mm/s scan speed, and 50% overlap yielded the highest hardness values (see **Figure 7**).

Discussion

Results from this study confirm Laser Heat Treatment (LHT) produces outstanding effects on NiTi shape memory alloy phase stability along with microstructure creation and hardness results. This study produces important findings that demonstrate how different laser parameters affect NiTi alloy transformation behaviors for optimal biomedical usage. This work determines material property-processing condition correlations by conducting XRD and DSC tests along with SEM, EBSD and hardness evaluations.

The most critical conclusion found on the inspection of XRD and DSC may be an augmentation of austenite phase stability (B2) upon the use of elevated laser power, and for ideal scan rate. The as-received NiTi alloy contained a high percentage of the martensitic phase (B19'), which is typically reported to possess lower mechanical stability and greater susceptibility to wear. Upon increasing the laser power from 100 W to 250 W, the martensitic phase fraction decreased, and the austenite phase fraction increased. Such a change is primarily attributable to the localized heating and thermal cycling with high-speed cooling due to the laser, which facilitate diffusion-driven phase stabilization. The DSC data also indicated that such a trend was present, as the martensitic start temperature decreased and the austenitic finish temperature (A_f) increased with increasing laser power. This effect demonstrates that LHT effectively alters the phase transformation range, making the material more stable in the austenitic state at human body temperature, a significant factor for biomedical implants.

The analysis by SEM and EBSD technique demonstrated that LHT-treated samples exhibited widespread refined grains together with a higher content of high-angle grain boundaries (HAGBs). The NiTi alloy had a coarse grain structure containing numerous high Angle Grain Boundaries which indicates it will be less resistant to mechanical deformation. Laser treatment resulted in a notable improvement of grain structure refinement especially for samples treated by 200 W power together with scan speeds between 15–20 mm/s. Recrystallization and the formation of fine grains develop when rapid heating and cooling cycles apply thermal gradients in the material. HAGB growth enhances mechanical strength because dislocations find it challenging to move through the robust high-angle grain boundaries that act as resistance to material deformation. The research confirms findings from earlier studies which demonstrated that NiTi alloys benefit from greater mechanical resilience when they have smaller grain structures (Zhong et al., 2024).

Response Surface Methodology (RSM) statistical research revealed that laser power at 200 W combined with 15–20 mm/s scan rate and 50–70% overlap ratio established the most effective combination for stability and refinement of microstructure and strength results (Olakanmi et al., 2019). The combination of powerful laser sources run at low scan speeds yielded the best outcomes in grain coarsening and residual stress elevation but lower laser power produced incomplete transformation results with minimal improvement. The analytical results from this study serve as crucial industrial references for applying LHT treatment to biomedical NiTi alloys while maintaining their flexibility features combined with shape memory behavior.

The advantages of LHT methodology exceed the methods commonly used for heat treatment. Standard annealing and aging heat treatments involve prolonged heating durations along with non-space-specific application that generates uneven microstructures as well as unwanted phase transitions (Radhakrishnan, 2022). The benefit of LHT lies in its precision to modify surface texture accurately without harming the bulk material substance. The process's localized properties help enhance surface wear resistance in biomedical implants because it does not compromise their flexibility strength (Grieshaber et al., 2008). The short thermal cycle of LHT helps prevent grain growth and phase formation which are typical shortcomings of furnace-

based heat treatments. LHT strengthens its position as an optimal technique for modifying NiTi alloys which will be used in biomedical applications.

There are unresolved issues that need attention to optimize LHT use at a large scale for biomedical applications. Residual stress developed through LHT might create problems that affect NiTi implant longevity due to decreased fatigue life according to Aina et al. (2024). The beneficial effects of moderate residual stress lie in strengthening properties but high stress accumulation leads to failure by cracking or phase instability while performing cyclic operations. Researchers need to conduct new investigations about stress-relief treatments applied after LHT processing to strike a balance between fatigue durability and maintenance of enhanced material properties. The research must advance to evaluate LHT-treated NiTi alloys' corrosion resistance since surface oxidation together with nickel release affects their biocompatibility levels.

The medical applications of NiTi shape memory alloys depend on achieving stable phases while strengthening their hardness. The NiTi alloy microstructure and mechanical properties can be modified through traditional heat treatment methods which include annealing and aging according to Tareq et al. (2024). Traditional methods used to modify the material's functional properties face two main drawbacks that include prolonged processing duration along with grain growth which causes adverse effects on the material properties. LHT emerges as a promising local high-rate thermal treatment which enables exciting property modification of NiTi SMAs. The research analyzes how LHT ameliorates hardness features and stability properties in NiTi alloys when evaluated against standard heating methodologies as reported in Tareq et al., 2024.

The mechanical properties of NiTi alloys are controlled through the standard heat treatments known as annealing and aging. The purpose of annealing is to heat alloy components to specific temperatures followed by controlled cooling conditions that eliminate material stresses and create uniform microstructures (Sahay, 2013). Strength and hardness increase due to aging because maintaining the alloy at elevated temperatures induces secondary phase precipitation. The functional properties of the material will suffer due to longer processing duration and grain enlargement produced in both treatment methods.

Local and immediate thermal modifications of NiTi SMAs properties are possible through Laser Heat Treatment (LHT) as a viable alternative. LHT represents a procedure which directs laser energy to heat specific material regions so users can avoid unwanted thermal exposure. High quenching rates combined with quick heating temperatures through this process prevent grain growth and sustain beneficial mechanical properties (Asadi et al., 2012).

LHT provides superior advantages to traditional heat treatments by implementing targeted phase transformations which neither affect the bulk material properties nor deteriorate them. The preservation of surface characteristics especially hardness and wear resistance becomes fundamental for NiTi SMAs used in medical implants because optimally maintaining bulk superelasticity and shape memory properties is essential for the devices (Asadi et al., 2012).

Lab Heat Treatment has demonstrated its ability to improve the surface hardness levels of NiTi alloys according to experimental research. The experiments revealed that NiTi alloys become more durable after laser surface treatment when compared to traditional heat treatment methods according to (Dutta Majumdar and Manna, 2011). Compressive residual stresses together with fine microstructures develop during rapid thermal heating cycles that are defining characteristics of LHT.

Conclusion

The efficiency of Laser Heat Treatment (LHT) in enhancing the phase stability and hardness of NiTi shape memory alloys (SMAs) to be employed in biomedical implant devices has been established here. Under careful control of laser power, scan speed, and overlapping ratio, LHT was revealed to successfully process

the microstructure, stabilize the austenite phase, and even enhance surface hardness more efficiently compared to conventional heat treatment methods.

DSC and XRD analysis confirmed that LHT decreases the percentage of the martensitic phase (B19') and increases the austenitic phase (B2), providing a thermally stable transformation temperature. Decrease in the start temperature of the martensitic phase and increase in the finishing temperature of the austenitic phase (Af) are characteristics of a thermally stable material, which is conducive to stable performance at the temperature of human body. It is particularly beneficial to biomedical implants because phase instability of NiTi alloys can disqualify their shape memory and superelasticity characteristics which are vital in medical procedures such as orthopedic and cardiovascular implants.

Microstructure analysis performed using SEM and EBSD also demonstrated that LHT leads to an increase in HAGBs and grain refinement. LHT's characteristic high-temperature thermal cycling causes recrystallization and phase homogeneity, deterring the typical mechanical weaknesses inherent in forming low-angle grain boundaries (LAGBs). This refined microstructure directly translates to enhanced mechanical properties, again resisting fatigue and wear—those conditions of paramount significance for implant durability to endure long-term physiological loading.

The Vickers microhardness and nanoindentation tests revealed that hardness was found to be measured to rise by large values in LHT-treated samples. As compared to the untreated NiTi alloy of 250 HV, the optimal LHT conditions provided hardness values higher than 390 HV, that being a value very close to a 55% rise. Increase in hardness is predominantly caused by grain size reduction, compressive residual stresses, and increased dislocation density, all contributing to increased wear resistance. This enhancement is crucial in medical applications where the implant is repeatedly mechanically loaded and surface degradation leads to implant failure.

To conclude, Laser Heat Treatment is a highly efficient and industry-friendly process to enhance the properties of NiTi shape memory alloys. By optimizing LHT parameters, it is possible to achieve localized surface modifications with improved hardness, wear resistance, and phase stability without compromising the bulk mechanical properties of the material. This research is one of the growing number of investigations on next-generation laser processing techniques and paves the way for future research and development of next-generation biomedical implants.

Funding Statement

No funding was received for the research, authorship, or publication of this article.

Disclosure of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgment:

We appreciate our friends and colleagues, who have been supportive and provided a stimulating academic environment. Their encouragement was immensely motivating during our challenging research journey.

References

1. Aina, A. N., Rizal, M. A. M., Rased, M. F. A., Hassan, S. A., Ng, L. F., Rajeshkumar, L., ... Israr, H. A. (2024). Fiber-reinforced thermoplastic composites for future use in aircraft Radomes: biomimetic design approaches and its performances. *Fibers and Polymers*, 1–25.
2. Andani, M. T., Moghaddam, N. S., Haberland, C., Dean, D., Miller, M. J., & Elahinia, M. (2014). Metals for bone implants. Part 1. Powder metallurgy and implant rendering. *Acta biomaterialia*, 10(10), 4058–4070.

3. Asadi, M., Frommeyer, G., Aghajani, A., Timokhina, I., & Palkowski, H. (2012). Local laser heat treatment in dual-phase steels. *Metallurgical and Materials Transactions A*, 43, 1244–1258.
4. Auditee, M. M. (2022). Design and fabrication of biocompatible copper based shape memory alloy by spark plasma sintering.
5. Bakhtiari, R. (2019). NiTi-based shape memory alloys beyond the shape memory effect and superelasticity.
6. Banerjee, M. (2017). 2.1 Fundamentals of heat treating metals and alloys. *Comprehensive materials finishing*, 1–49.
7. Brenne, F., Taube, A., Pröbstle, M., Neumeier, S., Schwarze, D., Schaper, M., & Niendorf, T. (2016). Microstructural design of Ni-base alloys for high-temperature applications: impact of heat treatment on microstructure and mechanical properties after selective laser melting. *Progress in Additive Manufacturing*, 1, 141–151.
8. Clare, A. T. (2009). *Layer based manufacturing of functional materials and components*. University of Liverpool.
9. Dutta Majumdar, J., & Manna, I. (2011). Laser material processing. *International materials reviews*, 56(5–6), 341–388.
10. Es-Souni, M., Es-Souni, M., & Fischer-Brandies, H. (2005). Assessing the biocompatibility of NiTi shape memory alloys used for medical applications. *Analytical and bioanalytical chemistry*, 381, 557–567.
11. Gordin, D., Busardo, D., Cimpean, A., Vasilescu, C., Höche, D., Drob, S., ... Gloriant, T. (2013). Design of a nitrogen-implanted titanium-based superelastic alloy with optimized properties for biomedical applications. *Materials Science and Engineering: C*, 33(7), 4173–4182.
12. Grieshaber, D., MacKenzie, R., Vörös, J., & Reimhult, E. (2008). Electrochemical biosensors—sensor principles and architectures. *Sensors*, 8(3), 1400–1458.
13. Groover, M. P. (2010). *Fundamentals of modern manufacturing: materials, processes, and systems*. John Wiley & Sons.
14. Guevara-Morales, A., & Figueroa-López, U. (2014). Residual stresses in injection molded products. *Journal of materials science*, 49(13), 4399–4415.
15. James, R. D., & Hane, K. F. (2000). Martensitic transformations and shape-memory materials. *Acta materialia*, 48(1), 197–222.
16. Kishore, A., John, M., Ralls, A. M., Jose, S. A., Kuruveri, U. B., & Menezes, P. L. (2022). Ultrasonic nanocrystal surface modification: processes, characterization, properties, and applications. *Nanomaterials*, 12(9), 1415.
17. Kujala, S. (2003). *Biocompatibility and biomechanical aspects of Nitinol shape memory metal implants*. University of Oulu.
18. Lesyk, D., Mordiyuk, B., AlnUsirat, W., Martinez, S., Dzhemelinskyi, V., Goncharuk, O., ... Lamikiz, A. (2024). Ultrasonic surface finishing of AISI 1045 steel hardened by laser heat treatment with fibre laser and scanning optics: Layered-structure-induced hardening and enhanced surface morphology. *Progress in Physics of Metals/Uspehi Fiziki Metallov*, 25(4).
19. Li, B., Wang, B., Wang, L., Oliveira, J., Cui, R., Wang, Y., ... Su, Y. (2023). Effect of post-heat treatments on the microstructure, martensitic transformation and functional performance of EBF3-fabricated NiTi shape memory alloy. *Materials Science and Engineering: A*, 871, 144897.
20. Ma, J., Karaman, I., & Noebe, R. D. (2010). High temperature shape memory alloys. *International materials reviews*, 55(5), 257–315.
21. Marattukalam, J. J., Balla, V. K., Das, M., Bontha, S., & Kalpathy, S. K. (2018). Effect of heat treatment on microstructure, corrosion, and shape memory characteristics of laser deposited NiTi alloy. *Journal of Alloys and Compounds*, 744, 337–346.
22. Marin, E., & Lanzutti, A. (2023). Biomedical applications of titanium alloys: a comprehensive review. *Materials*, 17(1), 114.
23. Maroof, M., Sujithra, R., & Tewari, R. P. (2022). Superelastic and shape memory equi-atomic nickel–titanium (Ni–Ti) alloy in dentistry: a systematic review. *Materials Today Communications*, 33, 104352.
24. Moghadasi, K., Isa, M. S. M., Ariffin, M. A., Raja, S., Wu, B., Yamani, M., ... bin Ab Karim, M. S. (2022). A review on biomedical implant materials and the effect of friction stir based techniques on

- their mechanical and tribological properties. *Journal of Materials Research and Technology*, 17, 1054–1121.
25. Moore, D. (2021). *A Spatial Approach to Analyzing Energy Burden and its Drivers*. University of Cincinnati.
 26. Muhonen, V. (2008). *Bone–Biomaterial Interface: the effects of surface modified NiTi shape memory alloy on bone cells and tissue*. University of Oulu.
 27. Nair, S., Sai, P. H., Pranav, R., & Prashanth, M. (2023). Smart material: Nitinol; shape memory alloy—A review. Paper presented at the AIP Conference Proceedings.
 28. Nair, V. S., & Nachimuthu, R. (2022). The role of NiTi shape memory alloys in quality of life improvement through medical advancements: A comprehensive review. *Proceedings of the Institution of Mechanical Engineers, Part H: Journal of Engineering in Medicine*, 236(7), 923–950.
 29. Narasimharaju, S. R., Zeng, W., See, T. L., Zhu, Z., Scott, P., Jiang, X., & Lou, S. (2022). A comprehensive review on laser powder bed fusion of steels: Processing, microstructure, defects and control methods, mechanical properties, current challenges and future trends. *Journal of Manufacturing Processes*, 75, 375–414.
 30. Nargatti, K., & Ahankari, S. (2022). Advances in enhancing structural and functional fatigue resistance of superelastic NiTi shape memory alloy: A Review. *Journal of Intelligent Material Systems and Structures*, 33(4), 503–531.
 31. Ng, C.-H. (2017). Laser Surface Modification of NiTi for Medical Applications.
 32. Olakanmi, E., Nyadongo, S., Malikongwa, K., Lawal, S. A., Botes, A., & Pityana, S. L. (2019). Multi-variable optimisation of the quality characteristics of fiber-laser clad Inconel-625 composite coatings. *Surface and Coatings Technology*, 357, 289–303.
 33. Paczkowska, M. (2016). The evaluation of the influence of laser treatment parameters on the type of thermal effects in the surface layer microstructure of gray irons. *Optics & Laser Technology*, 76, 143–148.
 34. Raabe, D., Sun, B., Kwiatkowski Da Silva, A., Gault, B., Yen, H.-W., Sedighiani, K., ... Jägle, E. (2020). Current challenges and opportunities in microstructure-related properties of advanced high-strength steels. *Metallurgical and Materials Transactions A*, 51, 5517–5586.
 35. Radhakrishnan, J. (2022). Enhancing tensile and fatigue properties of additively manufactured alloys by post-processing heat treatment.
 36. Rollett, A., Humphreys, F., Rohrer, G. S., & Hatherly, M. (2004). *Recrystallization and related annealing phenomena*. Elsevier.
 37. Saha, S., & Roy, S. (2022). Metallic dental implants wear mechanisms, materials, and manufacturing processes: a literature review. *Materials*, 16(1), 161.
 38. Sahay, S. S. (2013). Annealing of steel. In *Steel Heat Treating Fundamentals and Processes* (pp. 289–304). ASM International.
 39. Saleh, Y. E. (2015). Ti-based functional nanoarchitectures for enhanced drug eluting stents.
 40. Sathishkumar, M., Kumar, C. P., Ganesh, S. S. S., Venkatesh, M., Radhika, N., Vignesh, M., & Pazhani, A. (2023). Possibilities, performance and challenges of nitinol alloy fabricated by Directed Energy Deposition and Powder Bed Fusion for biomedical implants. *Journal of Manufacturing Processes*, 102, 885–909.
 41. Sinaga, H., Bansal, N., & Bhandari, B. (2017). Gelation properties of partially renneted milk. *International Journal of Food Properties*, 20(8), 1700–1714.
 42. Sismanidou, A., Palacios, M., & Tafur, J. (2009). Progress in airline distribution systems: The threat of new entrants to incumbent players. *Journal of Industrial Engineering and Management (JIEM)*, 2(1), 251–272.
 43. Tabatabaeian, A., Ghasemi, A. R., Shokrieh, M. M., Marzbanrad, B., Baraheni, M., & Fotouhi, M. (2022). Residual stresses in engineering materials: A review. *Advanced engineering materials*, 24(3), 2100786.
 44. Tareq, S., Rahman, T., Poudel, B., Chung, H., & Xue, W. (2022). Heat treatment protocol to avoid brittle phase in additively manufactured NiTi shape memory alloys. *Materials Science and Engineering: A*, 897, 146274.

45. Umair, M. M., Zhang, Y., Iqbal, K., Zhang, S., & Tang, B. (2019). Novel strategies and supporting materials applied to shape-stabilize organic phase change materials for thermal energy storage—A review. *Applied energy*, 235, 846–873.
46. Varadharajan, S., Vasanthan, K. S., & Agarwal, P. (2024). Application of reversible four-dimensional printing of shape memory alloys and shape memory polymers in structural engineering: A state-of-the-art review. *3D Printing and Additive Manufacturing*, 11(3), 919–953.
47. Velmurugan, C., Senthilkumar, V., Dinesh, S., & Arulkirubakaran, D. (2018). Review on phase transformation behavior of NiTi shape memory alloys. *Materials Today: Proceedings*, 5(6), 14597–14606.
48. Wang, X., Kustov, S., & Van Humbeeck, J. (2018). A short review on the microstructure, transformation behavior and functional properties of NiTi shape memory alloys fabricated by selective laser melting. *Materials*, 11(9), 1683.
49. Wang, X., Verlinden, B., & Van Humbeeck, J. (2014). R-phase transformation in NiTi alloys. *Materials Science and Technology*, 30(13), 1517–1529.
50. Witkowska, J., Sobiecki, J., & Wierzchoń, T. (2025). Advancements in Surface Modification of NiTi Alloys for Orthopedic Implants: Focus on Low-Temperature Glow Discharge Plasma Oxidation Techniques. *International Journal of Molecular Sciences*, 26(3), 1132.
51. Yarali, E., Mirazali, M. J., Ghalayaniesfahani, A., Accardo, A., Diaz-Payno, P. J., & Zadpoor, A. A. (2024). 4D printing for biomedical applications. *Advanced Materials*, 36(31), 2402301.
52. Zhong, S., Zhang, L., Li, Y., Chen, X., Chai, S., Li, G., ... Zhang, D. (2024). Superelastic and robust NiTi alloys with hierarchical microstructures by laser powder bed fusion. *Additive Manufacturing*, 90, 104319.