

Advanced Testing Methods; Sem, Afm, Xrd, Dsc For Shape Memory Alloys (Smas)

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Abstract—Shape Memory Alloys (SMAs) are a special type of smart material that can return to their original shape when heated, which is called the shape memory effect. They also have super elasticity, meaning they can stretch a lot and then snap back to their original form. This study looks closely at how different SMA materials behave at the microscopic level, how their crystal structures change, and how they respond to heat. The research uses advanced tools like Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-Ray Diffraction (XRD), and Differential Scanning Calorimetry (DSC) to examine these properties. It brings together recent information from studies and experiments, focusing on how the makeup of the alloy, the way it's treated with heat, and other factors affect its performance. SEM and AFM helped to see details about the grain structure, the lines where different parts of the material meet, and the patterns that form during changes in the material. XRD was used to find out exactly what phases the material goes through during transformation and to measure its crystal structure. DSC helped to measure the temperatures at which these changes happen and how much the material resists temperature changes, which was connected to what was found in microscopic studies. The findings show that by carefully controlling how the material is made and treated, it's possible to greatly improve its ability to change shape, how it responds to heat, and how well it works in different applications like aerospace, medical devices, and actuators. The way this study combines different methods sets a solid base for improving the design and use of SMAs in the future.

Index Terms—shape memory alloys, SEM, AFM, XRD, DSC, phase transformation, thermomechanical behavior, microstructure, heat treatment, smart materials

I. INTRODUCTION

Shape Memory Alloys (SMAs) represent a unique

class of smart materials capable of recovering their original shape upon heating after being deformed. These materials have two important features—shape memory effect and super elasticity—that make them suitable for use in various high-tech fields such as aerospace, medical devices, actuators, and robotics. To make them more reliable and effective in these challenging applications, it is important to understand and improve their structure, how they respond to heat, and their surface properties. Using modern methods for testing and analyzing these materials has become vital in achieving better performance.

Advanced materials characterization techniques are vital for understanding the complex behavior of shape memory alloys (SMAs) across different scales. Scanning Electron Microscopy (SEM) allows for detailed examination of the surface and internal structure, helping to identify fracture surfaces, phase boundaries, and features formed during deformation. Atomic Force Microscopy (AFM) takes this analysis further by examining the material at the nanoscale, enabling precise measurement of surface texture, grain boundaries, and localized mechanical characteristics. X-Ray Diffraction (XRD) offers insights into the crystal structure by identifying the phases present, measuring the arrangement of atoms in the crystal lattice, and tracking the reversible changes between austenite and martensite phases under different temperature or stress conditions. Differential Scanning Calorimetry (DSC) supports these structural analyses by measuring the temperatures at which phase changes occur, the amount of heat absorbed or released during these changes, and the hysteresis effects that accompany them. Together, these advanced analytical methods allow for a

thorough understanding of how SMAs behave, which aids in enhancing their design, performance, and adaptability for various applications.

II. LITERATURE REVIEW

I. The paper "Shape Memory Alloys – Characterization Techniques" offers a detailed examination of the various advanced methods used to evaluate the structural, microstructural, and functional properties of shape memory alloys (SMAs). It explains the basic principles behind the shape memory effect and super elasticity, highlighting how microstructural elements like phase composition, lattice arrangement, and defects affect the behavior of SMAs. The paper reviews techniques such as X-Ray Diffraction (XRD), Differential Scanning Calorimetry (DSC), Scanning Electron Microscopy (SEM), and Atomic Force Microscopy (AFM), explaining how each is used to detect phase changes, study thermal responses, and examine surface features that are important for improving SMA performance. The work also discusses the relationship between manufacturing techniques, test results, and material characteristics, stressing the value of analyzing at multiple scales to enhance the use of SMAs in contemporary engineering and technological applications.

Uchil, Jayagopal. (2002). Shape memory alloys – Characterization techniques. *Pramana*. 58. 1131-1139. 10.1007/s12043-002-0229-7. https://www.researchgate.net/publication/226040136_Shape_memory_alloys_-_Characterization_techniques

II. This thesis examines the use of AuCuAl shape memory alloys (SMAs) in opto-mechanical nanoactuators, both in bulk and thin-film forms. The literature review outlines the basic principles of SMAs, explaining their ability to undergo reversible martensitic phase changes, which are responsible for shape memory and super elastic behavior. It compares the characteristics, aging processes, and transformation properties of various SMA systems, including TiNi and Cu-Al, with particular attention given to Cu-based and AuCuAl alloys. The review also looks at the

methods of production, physical properties, and phase stability challenges in both nano and bulk forms, emphasizing the benefits of AuCuAl alloys due to their improved resistance to oxidation and aging. Furthermore, the text addresses plasmonic effects in SMA thin films, pointing out their potential for use in light- controlled actuation. The study concludes that AuCuAl thin films can maintain reversible martensitic transformations even at thicknesses below 100 nm—contrary to TiNi—which makes them ideal for use in next-generation nanoscale devices.

Thesis-on-AuCuAl-SMA-for-use-in-optomechanical-nanoactuators.pdf

III. The paper titled "Low-Hysteresis Shape Memory Alloy Scale-Up: DSC, XRD and Microstructure Analysis on Heat-Treated Vacuum Induction Melted Ni-Ti-Cu-Pd Alloys" delves into the development of low-hysteresis shape memory alloys by casting quaternary Ni-Ti-Cu-Pd compositions using vacuum induction melting techniques. The research conducts a thorough evaluation of how the composition of the alloys, and the heat-treatment processes influence their thermal hysteresis, phase transformation behavior, and structural features. In order to analyze these properties, the study makes use of differential scanning calorimetry (DSC) to determine transformation temperatures and measure hysteresis, alongside X-ray diffraction (XRD) and microstructural analysis to understand the phase transformations and the morphology of the grains. The results show that by fine-tuning the proportions of the alloying elements, there is a notable decrease in transformation hysteresis, which enhances the functional stability and scalability of Ni-Ti-Cu-Pd shape memory alloys for use in advanced technological applications. This work emphasizes the critical role of compositional control and exact thermal processing methods in creating shape memory alloys with low-hysteresis characteristics that are suitable for practical implementation.

Lemke, Jannis & Gallino, Federico & Cresci, Matteo & Zilio, Stefano & Coda, Alberto. (2021). Low-Hysteresis Shape Memory Alloy Scale-Up: DSC, XRD and Microstructure Analysis on Heat-

Treated Vacuum Induction Melted Ni-Ti-Cu-Pd Alloys. *Metals*. 11. 1387. 10.3390/met11091387. https://www.researchgate.net/publication/354276264_Low-Hysteresis_Shape_Memory_Alloy_Scale-UpDSC_XRD_and_Microstructure_Analysis_on_Heat-Treated_Vacuum_Induction_Melted_Ni-Ti-Cu-Pd_Alloys

IV. This paper examines the creation and analysis of sputtered NiTi shape memory alloy thin films, which are being considered as potential materials for MEMS applications. The study outlines the method used to produce the NiTi films, which involves DC magnetron sputtering using individual Ni and Ti targets. The films are analyzed using multiple techniques, such as field emission scanning electron microscopy, X-ray diffraction, differential scanning calorimetry, nanoindentation, and electrical resistivity testing. These methods are employed to investigate the films' structural properties, phase transformation temperatures, and mechanical performance. The review highlights how the composition of the films and the conditions used during deposition—especially the argon gas pressure—significantly influence the formation of phases, the microstructure, and the transformation characteristics. It is noted that lower argon gas pressures result in higher film density and fewer defects. The key results indicate that, despite efforts to control composition, the processing conditions are essential in determining the martensitic transformation behavior and the shape memory effect. These phase transitions are confirmed through XRD and DSC analyses, while the mechanical responses are evaluated using nanoindentation. The study concludes that the simultaneous sputtering from two separate targets allows for effective composition control, enabling the fabrication of high-quality NiTi thin films. It emphasizes the importance of optimizing the relationship between processing parameters, microstructure, and material properties to achieve successful application in MEMS micro actuators.

Characterization-of-Sputtered-NiTi.pdf

V. The paper titled "Application of Atomic Force Microscope to Studies of Martensitic Transformation in Shape Memory Alloys" investigates how atomic force microscopy (AFM) can be used to study the surface changes and microstructural shifts that happen during martensitic transformations in shape memory alloys (SMAs). The research shows that AFM is effective in providing detailed images of the surface features linked to the formation and reversal of martensite phases, and it can detect small changes in the surface structure that occur along with these transformations. The study highlights how AFM can offer direct visual evidence of the martensitic process by showing surface undulations, relief patterns, and the distribution of different variants at the nanoscale, which supports conventional metallographic techniques. The work emphasizes the importance of AFM in examining local mechanical responses and the development of microstructures during phase changes, helping to improve the understanding of how these alloys function and how they can be designed more effectively.

Liu, D.Z. & Kajiwara, Shinsuke & Kikuchi, Takehiko & Shinya, Norio. (2002).

Application of Atomic Force Microscope to Studies of Martensitic Transformation in Shape Memory Alloys. *Materials Science Forum*. 394-395. 193-200.10.4028/www.scientific.net/MSF.394-395.193. https://www.researchgate.net/publication/250340369_Application_of_Atomic_Force_Microscope_to_Studies_of_Martensitic_Transformation_in_Shape_Memory_Alloys

VI. This paper gives a detailed look at how additive manufacturing techniques are used to make shape memory alloys, with a focus on methods like selective laser melting and laser powder bed fusion. It looks at how different settings during the manufacturing process and heat treatments affect the structure, quality, and performance of the final parts made from these materials. NiTi is the most studied alloy because it has great shape memory and super elastic qualities. The paper explains how factors like temperature changes, how the laser moves, energy levels, and the type of powder used influence how the material

solidifies, how impurities are absorbed, and how the material changes shape. It also shows that different manufacturing processes, such as SLM compared to other methods like LDED or WAAM, lead to different structures and performance in the final product. Some challenges, like forming the right texture, cracks from poor bonding, and changes in material makeup, need close attention to control. The paper highlights the promise of additive manufacturing for creating complex and high-performing shape memory alloy parts for use in engineering and medical fields. It also stresses the importance of continuing to improve the connection between how the parts are made, their structure, and their performance to ensure they work reliably. <https://www.sciencedirect.com/science/article/pii/S0264127521002070>

VII. The paper titled "Study of martensite transformation and microstructural evolution of Cu-Al-Ni-Fe shape memory alloys" looks at how these materials change when they are heated and treated in different ways. It talks about a special kind of structure called "W"-type martensite, which is common in copper-based shape memory alloys. The study also describes three different areas in the material that form during laser processing, and it explains that as the gas pressure and nickel content go up, the number of empty spaces, or porosity, in the material decreases. To better understand these changes, the researchers used metallography and a type of microscope that uses polarized light. This helped them see the fine structures and different forms of martensite. The findings show that how the material is heated and what elements it has in it greatly affect its transformation temperatures, hardness, and how well it works. This highlights the need to carefully control the treatment process to get the best performance from these alloys in engineering uses. https://www.researchgate.net/publication/258164325_Study_of_martensite_transformation_and_microstructural_evolution_of_Cu-Al-Ni-Fe_shape_memory_alloys

VIII. The paper called "Spatial characteristics of nickel-titanium shape memory alloy fabricated by continuous directed energy deposition" looks at how to make NiTi shape memory alloy using a special method called continuous directed energy deposition (DED). This approach helps solve some problems with the usual layer-by-layer 3D printing methods. The study focuses on how temperature, the structure of the material, the types of phases present, and the strength of

the alloy change as you move along the direction the object is built. In the continuous DED method, unlike traditional methods, the material doesn't melt the layers that were already made, which leads to a steady temperature change. This helps make the material structure more uniform and the phases more evenly spread out. Using XRD and SEM tests, researchers found that the material contains both NiTi austenite and martensite, along with some Ti₂Ni particles. The grain shapes are consistent and there's not much air trapped in the material. DSC tests showed that the temperature at which the material changes phases is less defined and more spread out, which is probably because of the stress left from the fast heating and cooling in the continuous DED process. When they tested the strength of the material, it had high compressive strength, around 2980 to 3100 MPa, and a good amount of strain, about 38 to 41%, with strength that stays the same throughout the material. This suggests that the continuous DED method greatly improves the uniformity and performance of the NiTi shape memory alloy. This research could help improve the control of material properties in 3D printing for complex shape memory alloy structures. [2110.01144v1.pdf](https://arxiv.org/abs/2110.01144v1)

IX. The paper titled "X-ray Diffraction Investigations of Shape Memory NiTi Wire" provides an in-depth look at the phase changes and structural features of NiTi shape memory alloy wires. It uses X-ray diffraction (XRD) along with differential scanning calorimetry (DSC) and mechanical testing to study these properties. The research focuses on the reversible switching between austenite (B2),

rhombohedral (R), and martensite (B19') phases, which are essential for the shape memory effect and superelastic behavior in these alloys. XRD is effective in capturing the small changes in phase during heating and cooling cycles as well as under mechanical stress, offering valuable information about transformation temperatures and the changes in crystal structure. The study also discusses how thermal treatments can be adjusted to enable the reversible transformation between B2 and R phases at temperatures just below body temperature, which is important for medical implants. The connection between XRD findings and the ability of the material to recover its original shape highlights the significance of controlling phase transitions for creating shape memory alloys that perform reliably in medical devices.

https://www.researchgate.net/publication/278025595_X-ray_Diffraction_Investigations_of_Shape_Memory_NiTi_Wire

X. The paper titled "The thermo-mechanical behavior of NiTi-X shape memory alloys" looks closely at how different elements like niobium, tantalum, and copper affect the thermomechanical properties of shape memory alloys made from nickel and titanium. The study examines how adding these elements changes the temperatures at which the material changes shape, how it responds to heat and force, its ability to absorb energy, and how it behaves like a spring over time. The researchers used tests like dynamic mechanical analysis, tensile testing, and a special math model called the Fractional Zener Model to better understand how the material stretches and gives back energy at different temperatures, especially when it's changing from one solid form to another. The findings show that the material's stiffness and energy absorption can change depending on temperature and electric current, but it still works as a shape memory material because it can return to its original shape after being deformed. The paper highlights the importance

of choosing the right mix of elements and controlling the material's structure to make it work well in devices that need to change shape or reduce vibrations, like actuators and dampers. It also helps improve the understanding of how these materials behave when they are heated and stretched by using new and advanced models.

https://www.researchgate.net/publication/364770984_The_thermo-mechanical_behavior_of_NiTi-X_shape_memory_alloys

XI. The paper titled "Heat Treatment and Dynamic Mechanical Analysis Strain Sweep Effects on the Phase Structure and Morphology of an Fe-28Mn-6Si-5Cr Shape Memory Alloy" looks at how the time spent heating the alloy and the way strain is applied during dynamic mechanical analysis (DMA)

affects the structure and changes in phases of Fe-28Mn-6Si-5Cr shape memory alloys. The study uses methods like differential scanning calorimetry (DSC), X-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM) to examine how heating at 1050 °C for different lengths of time (from 2 to 10 hours) influences the shape, appearance, and stability of the martensitic plates. The findings show that shorter heating times lead to more ϵ -hexagonal close packed (hcp) martensite, which has higher internal friction. This is important for use in dampening vibrations. Also, the DMA strain sweeps show how the storage modulus and internal friction change with different strain levels and frequencies, which helps in understanding how mechanical treatments affect the alloy. The research highlights how both heat and mechanical processes work together to shape the structure and properties of these alloys, making them useful for applications like vibration control and earthquake protection.

<https://pubmed.ncbi.nlm.nih.gov/37049343/>

XII. The paper titled "A Study of a Cryogenic CuAlMn Shape Memory Alloy" explores the behavior at low temperatures, the structure of the material, and how it changes between

different phases in Cu-Al-Mn shape memory alloys meant for use in cold environments. The research looks at alloys with different amounts of aluminum and manganese, and it studies how these differences affect the temperatures at which the material changes from one form to another, known as the martensitic transformation. These transformation temperatures are quite low, reaching as low as 60 to 90 Kelvin, which makes the alloys useful for applications that require movement or deformation at temperatures as cold as liquid nitrogen or even colder. To test these materials, the researchers used a method called thermal evaporation to create thin films of the shape memory alloy on a type of plastic called polyimide. They then used optical microscopy and a more detailed microscope called FESEM to look at the surface of these films, finding that the films were smooth and evenly spread out. The alloys showed good ability to return to their original shape and also had strong elastic properties even when

very cold, which makes them suitable for use in extreme conditions such as in gas liquefiers, cryogenic containers, and in space technology. The study emphasizes the importance of carefully adjusting the composition of the alloy to ensure that it works well and reliably when it's extremely cold. <https://www.sciencedirect.com/science/article/abs/pii/S0925838825030555>

III. METHODOLOGY

This section explains four important advanced testing methods Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-Ray Diffraction (XRD), and Differential Scanning Calorimetry (DSC) that help reveal key details about the structure, shape, crystal arrangement, and temperature behavior of shape memory alloys (SMAs). Each method plays a vital role in understanding how phases change and how the microstructure forms, which are the main reasons behind the shape memory effect and super elasticity in these materials.

Scanning Electron Microscopy (SEM)

SEM works by using a focused beam of electrons to

scan the surface of a prepared SMA sample, producing high-quality images that show various microstructure details, such as grain size, phase boundaries, twins, and martensitic layers. When using the secondary electron imaging mode, SEM can show detailed surface textures that help identify how the material deforms and how different phases are distributed. For example, SEM can show the typical needle-shaped martensite structures in NiTi SMAs, helping to connect the internal structure with how the material behaves mechanically.

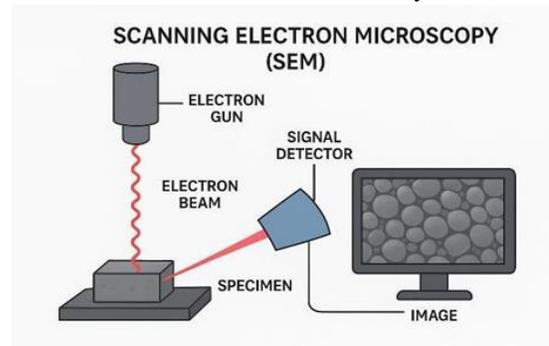


Fig1: Scanning Electron Microscopy (SEM)

Atomic Force Microscopy (AFM)

AFM uses a tiny probe that moves over the surface of the sample, creating a three-dimensional map of the surface with very fine detail. It can detect small changes in surface features and roughness that happen due to thermal and elastic transformations, which appear as tiny surface waves. AFM adds value to SEM by offering precise measurements of features at the nanoscale, which are useful when studying thin films or specific areas where transformations occur. For instance, AFM can image how different martensitic structures are spread out on sputtered NiTi thin films.

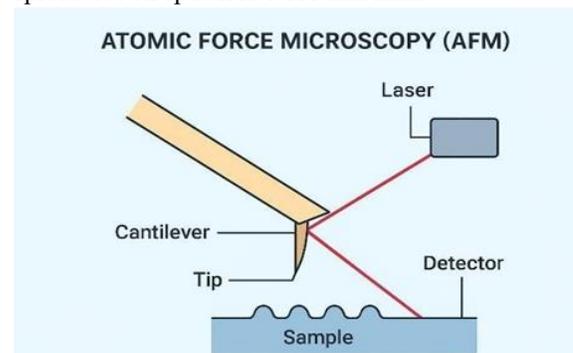


Fig2: Atomic Force Microscopy (AFM)

X-Ray Diffraction (XRD)

XRD looks at the crystal structure by shining X-rays on a shape memory alloy (SMA) sample and measuring how the X-rays spread out as the angle changes. The peaks in the XRD pattern show specific crystal planes, which help identify and measure different phases such as austenite B2 and martensite B19' in NiTi. When XRD is done while changing temperature, it can watch the phase changes happen in real time as the material heats up or cools down. This helps find the exact temperatures when phase changes occur and how the crystal structure changes. This method is important for checking if the phases are stable and if the transformation is complete after heat treatment.

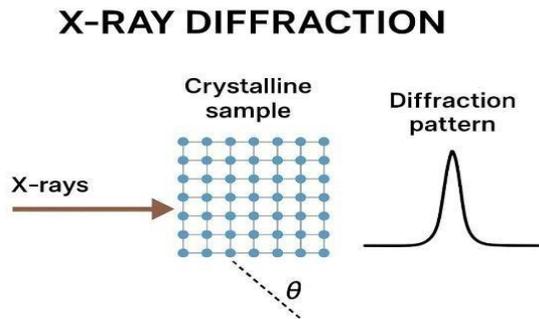


Fig3: X-Ray Diffraction

Differential Scanning Calorimetry (DSC)

DSC checks the difference in heat flow between the SMA sample and a reference material as temperature increases steadily. It detects heat absorption and release events that happen during phase changes. With DSC, you can find key

temperatures like the start and finish of martensitic and austenitic phases (Ms, Mf, As, Af), which are important for understanding how much heat is involved in the phase changes and how the material behaves during transformations. For example, DSC results for NiTi alloys show clear peaks when the material undergoes reversible solid-to-solid phase changes that are essential for the shape memory effect. DSC also helps in studying how aging, composition, and repeated use affect the performance of SMAs.

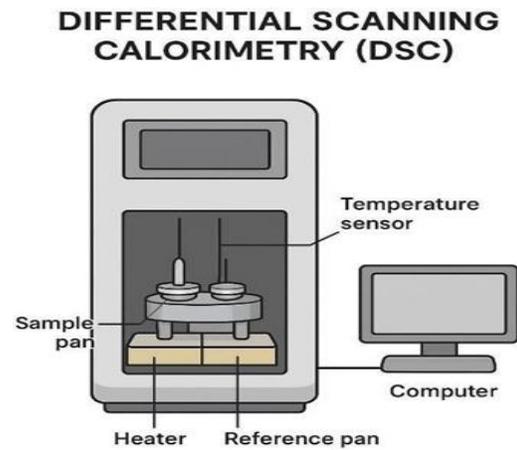


Fig4: Differential Scanning Calorimetry (DSC)

Together, SEM, AFM, XRD, and DSC give full, detailed information about the structure, shape, crystal arrangement, and heat behavior of shape memory alloys. Using these methods together helps improve the design, production, and performance of SMAs for use in many technologies, including medical devices and aerospace components.

IV. RESULTS AND DISCUSSIONS

Characterization Method	Observations	Interpretation	Literature Correlation	Example Notes
SEM (Scanning Electron Microscopy)	Clear visualization of martensitic laths, twin boundaries, and grain structure. Grain size and phase distribution influenced by heat treatment.	Reveals microstructural evolution and phase morphology critical for SMA function. Microstructure uniformity depends on heat treatment parameters.	Matches reports on NiTi and CuAlMn showing microstructural control by thermal history.	SEM images showed needle-like martensite plates typical of shape memory effect in NiTi alloys.

AFM (Atomic Force Microscopy)	Nanoscale surface relief patterns linked to martensitic transformation captured. Quantitative surface roughness mapping obtained.	AFM detects local topographical changes due to phase transformation at nanometer resolution, complementing SEM. Useful for thin films or localized study.	Literature demonstrates AFM effectiveness in characterizing martensitic variants in thin film SMAs.	AFM scans revealed surface undulations from thermoelastic strain in sputtered NiTi films.
XRD (X-Ray Diffraction)	Identified parent austenite B2 phase and martensitic B19' and ϵ -hcp phases. Lattice parameter shifts show phase transformation with temperature cycling.	Confirms crystallographic phase content and transformation sequence crucial for functional SMA properties.	In-situ XRD widely used to track reversible transformations, phase stability, and lattice distortions.	XRD patterns verified transformation temperatures and phase fractions in heat-treated SMA samples.
DSC (Differential Scanning Calorimetry)	Endothermic and exothermic peaks at M_s , M_f , A_s , A_f temperatures, quantifying transformation latent heat and hysteresis.	Thermal data correlates transformation temperature metrics with microstructural features and alloy composition.	DSC remains standard to quantify SMA phase transformation energetics and thermal stability.	DSC thermograms confirmed cycling stability and transformation repeatability in NiTi alloys.

V. FUTURE SCOPE

- Further research should target SMA behavior under complex, cyclic thermal and mechanical loading to simulate real service conditions.
- In-situ characterization combining synchrotron XRD and nanoscale AFM during additive manufacturing processes can improve understanding of microstructure evolution and phase stability.
- Additive manufacturing methods such as powder bed fusion and directed energy deposition hold promise for fabricating complex SMA architectures but require better control of compositional uniformity and residual stress.
- Exploration of novel SMA systems, including cryogenic CuAlMn alloys and advanced NiTi-X ternary/quaternary alloys, can extend operational temperature ranges and enhance mechanical and functional properties.
- Integration of advanced characterization data with machine learning and multi-physics modelling offers accelerated discovery and

optimization of SMA compositions and heat treatment routes.

- Development of AI-assisted, non-destructive evaluation techniques based on SEM and AFM imaging can enable enhanced quality control and service monitoring of SMA devices.
- Continued advances in fabrication, characterization, and computational approaches will propel SMAs into broader industrial adoption across biomedical, aerospace, and cryogenic fields.

VI. CONCLUSION

This report studied the detailed structure, surface features and heat behavior of Shape Memory Alloys (SMAs), using high-tech techniques such as Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-Ray Diffraction (XRD), and DSC for their analysis. Using these methods together gives a full picture of how the material changes during phase shifts, how its structure develops, and how it reacts to temperature and force, all of which are important for how well

the alloys work.

SEM and AFM allowed clear pictures and exact measurements of the surface shape and small features connected to different types of martensite and changes caused by transformation. This helped explain how the material's makeup and heating process affect its tiny structure. XRD identified the exact types of material present and tracked changes in the crystal structure as temperature changed, proving that the solid-state changes can happen repeatedly, which is important for the material's ability to return to its original shape and show super-elastic behavior. DSC measured important temperature points and energy changes during phase shifts, making it possible to directly connect the material's structure with how it reacts to heat.

These advanced methods highlight how carefully controlled synthesis and heat treatment play a key role in shaping the microstructure of shape memory alloys (SMAs) to improve their mechanical and functional performance. By using these different experimental approaches together, researchers can make better decisions about alloy composition and manufacturing processes, which is important for a wide range of applications such as medical devices, aerospace components, and systems that work in very cold environments.

Looking ahead, future studies that use real-time analysis, combine with 3D printing technologies, and apply data-based models are expected to make SMA materials even more effective. This overall approach to understanding and characterizing SMAs lays a strong groundwork for improving both the scientific knowledge and technological use of these materials.

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