Material Characterization of Shape Memory Alloys Using SEM, XRD, and Chemical Composition Analysis for Smart Engineering Applications

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Abstract

Shape Memory Alloys (SMAs) are advanced materials with the ability to retain their initial form upon heating due to their unique shape memory effect (SME) and pseudo elasticity due to the phase transformations of martensitic to austenitic.This memory effect originates from the reversible phase changes of austenite and martensite crystalline structures. These alloys are biocompatible, that is, they do not cause any kind of reaction in the human body; they are lightweight, high damping capacity, corrosion-resistant, stability and incorporate a superior thermal sensitivity. This study concentrated on the microstructural and phase characteristics of SMAs using SEM, XRD, and EDS for chemical composition analysis. SEM analysis reveals surface morphology and martensitic structures, while XRD confirms the presence of both martensite and austenite phases. Chemical composition testing validates the alloy's purity and elemental distribution. SMAs have revolutionized applications in civil engineering, biomedical, industrial, aerospace, automotive, and robotics fields. This articles provide a comprehensive review on material behavior, which is necessary for engineering applications.

Key words: Shape Memory Alloys (SMAs), NiTi Alloy (Nitinol), Shape Memory Effect (SME), SEM, XRD, EDS, Biomedical, Aerospace.

1. Introduction

Shape Memory Alloys (SMA) are self-sensing materials that can return to their initial shape again when they are heated at a suitable temperature. The study and development of SMA began in the mid-20th century. In 1932, Arne Olander first called the shape memory alloys (SMAs) "smart alloys." In 1941, Vernon first called the dental polymer "shape-smooth." [1, 3]. In the 1960s, the American Naval Research Laboratory developed nitinol. NiTi revolutionized the field of SMA because of SMA's enhanced shape memory effect, flexibility, and optimized thermal response. Recently, biomedical, consumer products, fashion, robotics, automotive, and aerospace increased in areas adopting SMA. [1,2]. The crystal structures show a reversible phase transition between austenite and martensite, resulting in this effect, as depicted in **Fig.1.**

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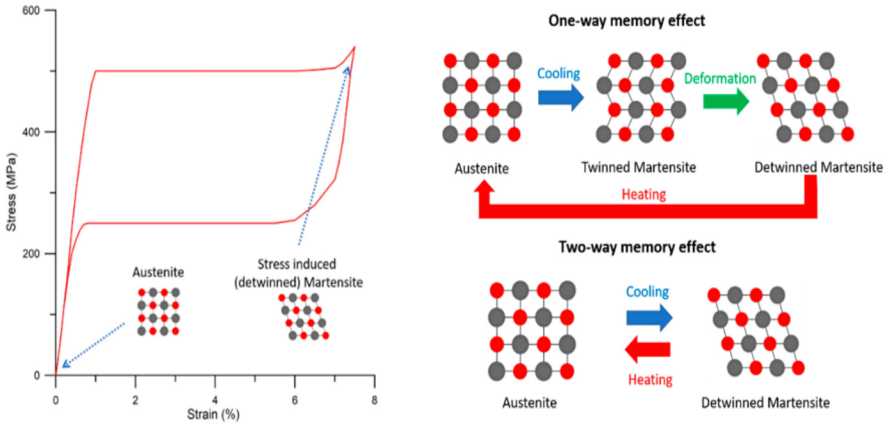

Fig.1 Mechanisms in the SMA [1]

Shape Memory Alloys (SMAs) represent a unique class of advanced functional materials that exhibit distinctive thermomechanical features, including the Shape Memory Effect (SME) and superelasticity.These phenomena are primarily attributed to reversible martensitic-austenitic phase transformations, which allow the material to recover its pre-deformed shape upon heating or mechanical unloading. The excellent properties of nickel-titanium (NiTi or Nitinol) in various SMAs, such as high corrosion resistance, high biocompatibility, low density, and high fatigue strength, have generated considerable discussion. The increasing demand for intelligent engineering materials in important sectors, for instance,biomedical implants, aerospace actuators, automotive safety systems, industry and consumer products, civil infrastructure, and robotics, has led to extensive research on SMAs. However, the functional performance of SMAs is strongly governed by their microstructure, phase composition, and chemical homogeneity, which necessitates precise characterization.

The advancedmaterial characteristics techniques play an important role in achieving these objectives.Electronmicroscopy (SEM) explains detailed insights into the scanning, surfacemorphology, and martensiticstructures.X-ray diffraction (XRD) helps in identifying and confirming the crystalline stages of martensite and austenite, and energy dispersive X-ray spectroscopy gives the exact chemical characteristics of themixture. Together, all these methodsprovide a comprehensive understandingof the structural and chemical properties of SMA.

2. Techniques and Procedures:

The material property evaluation of shape memory alloys, the sample preparation method involved the following steps and shown in figure 2(a) to 2(c):

2.1 Sample Preparation:

A specimen of Shape Memory Alloy (SMA) was sourced with dimensions of 100 mm in length and 12 mm in diameter.

The specimen was sectioned into smaller pieces of 3 mm thickness using a Wire EDM process.

The cut pieces were polished using different grades of emery paper with varying grit sizes,followed by polishing with diamond paste.

After polishing, the sample underwent an etching operation using a solution of HNO_3 (3%) (Nitric acid), HF (2%) (hydrofluoric acid), and rest deionized water.

Fig.2 (a).Wire EDM

Fig.2 (b).NiTi Sample

Fig. 2(c).NiTi Sample after cutting and polishing

2.2 Chemical Composition Analysis: As shown in Table 1, the EDS results revealed an elemental composition of approximately Ni 50.2% and Ti 49.8%, indicating a near-equiatomicNiTi alloy with high purity and minimal contamination, as trace elements such as oxygen and carbon were detected in negligible quantities. In Figure 3(a) presents the elemental composition of the NiTi alloy and shows the nickel and titanium are the major constituents and Figure 3(b) illustrates the EDS spectrum of the NiTi sample, providing confirmation of the high purity of the alloy and near equiatomic composition.

Table: 1. Elemental Composition of NiTi-Based Shape Memory Alloy.

Chemical Properties						
Ni	**Ti**	**Si**	**S**	**Fe**	**Zr**	**Mo**
55.10	**43.70**	**0.904**	**0.248**	**0.036**	**0.002**	**0.003**

El	%	+/- 3σ
Si	0.904	0.062
S	0.248	0.012
Ti	43.70	0.18
Fe	0.036	0.010
Ni	55.10	0.17
Zr	0.002	0.001
Mo	0.003	0.001

Fig.3 (a) EDS spectrum showing elemental composition of NiTi alloy.

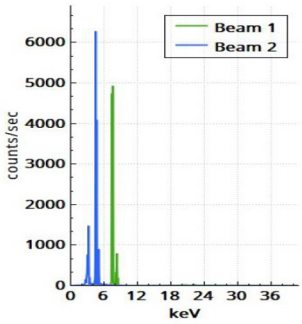

Fig.3 (b) EDS Spectrum of NiTi Sample.

3. Material Characterization: Material Characterization plays a key role in understanding the basic concepts of chemical properties, micro and crystal structure. In the present study, different cutting edge techniques for example EDS, XRD and SEM have been used for comprehensive study.

3.1Energy Dispersive Spectroscopy (EDS/EDX):EDS analysis determining the elemental composition and spectrum of NiTi in sample.In sample result, spectrumBeam 2 (blue spectrum) exhibits a dominant peak at approximately 6.4 keV, corresponding to the Fe Kα line, with a maximum intensity of 6200 counts/sec. This indicates that Beam 2 is highly sensitive to iron-rich phases within the sample. In addition to this primary peak, smaller features are visible between 5 and 7 keV, which can be attributed to elements such as Mn (Kα -5.9 keV) and possibly trace contributions from Cr (Kα ~5.4 keV). These results suggest that Beam 2 provides strong excitation for medium atomic number elements. [4-5].

Beam 1 (green spectrum), on the other hand, shows a pronounced peak at approximately 8.0 keV, which matches the Cu Kα line, and an additional broader peak at 11 keV that may correspond to the Ni Kβ (8.3 keV) and/or contributions from higher-energy transitions of heavier elements. The maximum intensity of this peak is ~4800 counts/sec. The absence of significant low-energy peaks indicates that Beam 1 is primarily effective in detecting heavier elements compared to Beam 2.The comparison of both spectra reveals a complementary detection profile. Beam 2 is efficient for detecting lighter and medium atomic number elements (Cr, Mn, Fe), while Beam 1 enhances the sensitivity toward heavier elements (Ni, Cu). This complementary response can be attributed to differences in beam excitation conditions, including incident energy, beam–sample interaction volume, and penetration depth.
The elemental identification also confirms that the analyzed material exhibits a heterogeneous microstructure, consisting of both Fe-rich and Cu/Ni-rich phases. Such multiphase distribution is typical in engineering alloys and composite systems, where Fe contributes to the structural backbone, while Ni and Cu enhance functional and mechanical properties. Similar observations have been reported in alloy characterization studies using EDS, where multi-beam excitation improved phase identification and minimized elemental detection bias [5-6].

3.2Scanning Electron Microscopy (SEM**):**Scanning Electron Microscopy (SEM) is anessential technique for studying surface morphology, microstructure features, and phase distributions of metallic and composite materials.

3.2.1. Micro structural Features: The image as shown in Figure 4 has been successfully scanned by scanning electron microscopy to analyze the microstructure qualities, for example, cracks, microholes, globules, andglobules, and boundaries. The microstructuralanalysis shows that the material features clearly defined the crystalline structure, as shown by visible grain boundaries in the SEM image.Such boundaries act as barriers to dislocation motion and affect strength, ductility, and creep resistance, which are important in determining mechanical behavior. The dark areas observed within microstructures are like pores or inclusions due to solidification, processing, or trapped gases. These properties are detrimental to performance because they can become stress concentrators and starting points for cracks, reducing lifespan and overall reliability. Also, the presence of fibrous microstructure in alloys undergoing rapid cooling or thermal treatment indicates solidification structures or phase change. These layered shapes often contribute to increasing the mechanical properties by refining the shape of the granules and increasing their stiffness, but too much separation can also cause unevenness in the properties. [6-7]

3.2.2 Pore & Particle Analysis: The distribution of pores within the microstructure appears heterogeneous, suggesting non-uniform solidification or localized variations in heat transfer during processing. Pores are present in different morphologies, where some are globular or rounded, typically associated with gas entrapment or solidification shrinkage under near-isotropic conditions. In contrast, elongated pores are also visible, which may arise from anisotropic shrinkage stresses or directional solidification, indicating that the pore formation mechanism is not uniform across the sample. Such anisotropy in pore geometry can strongly influence the mechanical integrity of the material, as elongated pores are often more detrimental due to stress concentration effects compared to spherical pores. Furthermore, the pore size exhibits a wide range, from sub-micron levels up to several microns, as confirmed by the scale bar of 30μm. This variation in size distribution highlights the coexistence of fine micro-porosity along with relatively larger voids, which together can significantly affect density, strength, and fatigue resistance. The heterogeneous nature, morphology, and scale of pores therefore provide important insights into the solidification dynamics and potential defects in the manufacturing process. [8, 9, 11].

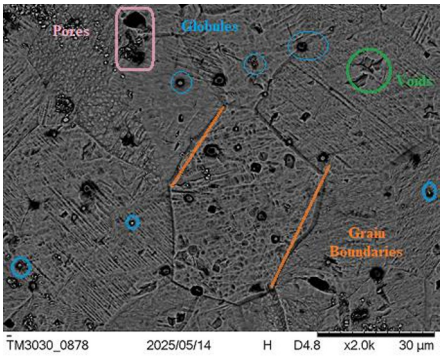

Fig.4.SEM microstructure of NiTi alloy.

3.3 X-ray Diffraction (XRD): This is powerful techniques that provides detailed information of structural features and phase transformation as shown in Figure 5. In this investigation XRD pattern spans the range of 20° to 80°.The most intense diffraction peak appears at approximately ($2\theta \approx 43°$) with a very high intensity (8200 a.u.), indicating the highly crystalline nature of the material as evidenced by the sharp and well-defined peaks. The strongest reflection around 43° is characteristic of metallic phases with an FCC structure, such as Ni or Cu. Additional minor peaks are observed near $2\theta \approx 77°$, and a weaker reflection may also be present at lower angles (38°). The dominance of a single strong peak with very few weak reflections suggests that the material is predominantly single-phase with high phase purity, while the presence of minor peaks may indicate limited contamination or slight phase transformations.[8,10-11].

In the case of NiTi shape memory alloys (SMAs), the sharp reflection near 43° is typically associated with the B2 austenite phase, specifically corresponding to the (110) crystallographic plane. The B2 phase is stable at room temperature and is responsible for the shape memory and super elasticbehavior of NiTi alloys. It should also be noted that the (111) reflection of FCC Cu occurs at a similar position, which may overlap with the NiTi B2 peak, suggesting a possible composite signal if copper is present in the sample.

Overall, the sharpness and intensity of the peaks confirm that the sample is highly crystalline with negligible amorphous content. The obtained XRD pattern provides clear evidence of the prevailing phase, whether austenite (B2) or martensite (B19′), in NiTi SMAs. However, precise phase identification requires further peak indexing, assignment of the corresponding crystallographic planes (hkl), and complementary compositional analysis. [8, 12-14].

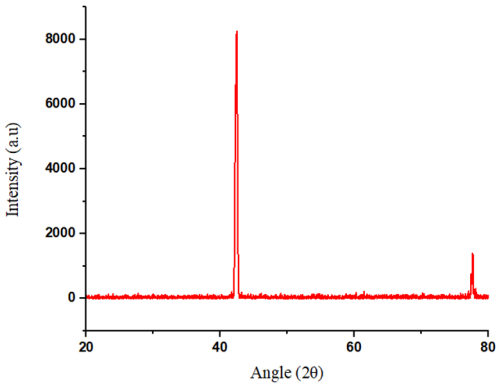

Fig.5. XRD pattern of NiTi alloy

Conclusions

The characterization of Shape Memory Alloys (SMAs) using SEM, XRD, and EDS provided comprehensive insights into their microstructural and chemical properties. SEM analysis confirmed the presence of martensitic variants along with detailed surface morphology, indicating phase transformation behavior. XRD patterns validated the coexistence of austenite and martensite phases, which are fundamental to the shape memory effect and pseudoelasticity. Furthermore, EDS analysis verified the elemental distribution and alloy purity, establishing the suitability of SMAs, particularly NiTi, for biomedical and aerospace applications. Overall, the results reaffirm that the functional reliability of SMAs is strongly governed by their microstructural stability and compositional uniformity.

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