Influence of applied stress on shape memory characteristics of $Ni_{50}Ti_{45}Cu_{5}$ (at.%) alloy subjected to thermomechanical cycling

Swaminathan G.[1,*], Sampath V.[2], Adarsh S.H[3].

[1]Department of Mechanical Engineering, PSG Institute of Technology and Applied Research, Neelambur, Coimbatore - 641 062, India.

[2] Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai 600 036, India.

[3]Department of Mechanical Engineering, Siddaganga Institute of Technology, Tumakuru – 572103, India.

*E-mail: swamioff@gmail.com

Abstract

Shape memory alloys (SMAs) have made rapid progress into many domains, primarily biomedical (endovascular stents, orthodontic archwires), and engineering (smart actuators, robotics, hydraulic couplings). The selection of an SMA for the indented application is based on its characteristic phase transformation temperatures. These characteristic temperatures are influenced by myriad parameters, such as composition, microstructure of the alloy, defect density, etc. When an SMA under an external load is subjected to cyclic operations to perform useful work, for example, actuators, these characteristic temperatures are modified. This study, therefore, aims to understand the influence of external loading on the shape memory characteristics of an $Ni_{50}Ti_{45}Cu_{5}$ (at.%) alloy. A wire of 1.43 mm diameter and length 100 mm was subjected to heating and cooling between its phase transformation temperatures in a cyclic manner under constant stress (of up to 60 MPa). The maximum recovery strain, actuation/retraction rate, and the stress influence coefficient (SIC) were determined and compared with those of the other Ni-Ti and Cu-based SMAs. The results show that the raising the load level causes an increase in the transition temperatures, especially the M_s (martensite start temperature) rather than the other phase transformation temperatures (martensite finish (M_f), austenite start (A_s), austenite finish

(A_f)). It also significantly affects the recovery strain and the rate of retraction during forward transformation and the symmetry of operation.

Keywords: NiTiCu SMA, functional fatigue, stress influence coefficient, actuation/retraction rate.

1. Introduction

Near-equiatomic NiTi, commercially known as Nitinol, is one among the most useful commercial shape memory materials till date owing to its excellent functional properties, together with its good mechanical properties and biocompatibility. A unique way to recover a large strain in SMAs is by either shape memory effect (heating) or superelastic effect (loading/unloading) [1–3]. These effects originate from the solid-state, diffusionless martensitic phase transformation [4]. During the deformation of the martensitic phase, the strain is accommodated by twinning rather than slip which accounts for the reversibility. The necessary condition for a material, which undergoes a martensitic transformation, to show the shape memory effect is the symmetry of austenite should be higher than that of the product phase (martensite), and the point symmetry group of the martensite phase must fall within the parent phase symmetry group [5,6]. There are other criteria that enhance the SME and SE, such as the strength of the transforming phases, morphology of martensite, and level of ordering of the austenite phase [6].

Both superelasticity (photodiodes, orthodontic archwires, eyeglass frames, hydraulic couplings, and civil structures) and shape memory effect (robotic arm grippers, stents, switches, and smart actuators) are deployed from the applications point of view [7–11]. The choice of the alloy mainly depends on the hysteresis width and phase transformation temperatures. The main advantage of Ni-Ti SMAs is the ability to modify the transformation temperatures just by controlling the composition of the alloys and their thermomechanical treatments. When the Ni content of the binary alloy exceeds 50.2 at.%, its transformation temperatures decrease at the rate of 10°C per 0.1 at. % of Ni increase [12–

[14]. This makes the alloy suitable for low-temperature and room temperature applications, where SE is needed. In contrast, the alloy is less sensitive to the variation in Ti content on the Ti-rich side, and the characteristic transition temperatures are pushed to above the ambient temperature, which makes the alloy suitable for applications that demand SME. The functional properties of the SMAs, such as recovery strain, hysteresis width, etc. are affected when SMAs are subjected to thermal or thermomechanical cycling between their characteristic phase transformation temperatures. This change of shape memory characteristics by the repeated cyclic operation is called functional fatigue of SMAs [15,16]. During thermomechanical cycling, it is observed that dislocations are generated [17], the microstructure of the alloy gets altered [18], and fine precipitate particles [19] are formed. The occurrence of functional fatigue is attributed to these microstructural changes.

Among these microstructural features, the generation of dislocation is the primary contributing factor for the changes in functional fatigue properties. This is attributed to the stress field created by the generation of dislocation [20] that affects the transformation of phases either by preventing or facilitating the austenite/ martensite interface movement during heating or cooling. Generally, thermal cycling tends to reduce the phase transformation temperatures by hindering the motion of the austenite/martensite interface by the dislocations that are generated during the cycling. On the other hand, thermomechanical cycling increases the transformation temperatures.

Ibarra et al. [21] observed two groups of dislocations that are generated during cycling. One group hinders the martensitic transformation, while the other facilitates it and leads to either postponing or advancing the transformation. This tends to affect the performance of the device as it will respond to the stimulus (temperature) at different temperatures as compared to the intended temperature. Smart actuators made of shape memory alloys and the applications, such as coronary stents and aortic valve, are expected to have a lifespan of more than 10 million cycles [17,22,23]. Under these conditions, these devices are more likely to undergo functional fatigue, and as a result, these devices lose their intended functionality/performance.

The fatigue life of a component/device depends mainly on the alloy composition, microstructure, applied stress level, magnitude of strain, temperature cycle range, etc. Among these, stress is considered to be a major parameter, which affects the fatigue life [24,25]. Moreover, most devices use Ni-Ti SMAs. Hence the basic alloy composition is fixed. Therefore, a ternary addition to Ni-Ti would be of interest in improving fatigue life. In this context, Cu addition could serve the purpose as they have some distinct advantages. The critical transition temperatures of binary Ni-Ti SMAs, especially Ni-rich alloys, where even the variation of the Ni content by as low as 0.1 at. % lowers the phase transition temperatures by 10 ℃. This, therefore, necessitates a closer control over the composition. The addition of copper as a ternary addition overcomes this compositional sensitivity caused by Ni in Ni-Ti SMAs. Moreover, copper addition reduces the transformation hysteresis width, which is an essential requirement for the actuator applications, as a smaller hysteresis width decreases the response time of the actuator [26,27].

In addition, the effect of applied stress on transformation temperatures is significant and it can be described using Clausius–Clapeyron equation. Generally, an increase in the applied stress level increases transformation temperatures [12]. When SMAs are used in the actuator applications, they are subjected to operate at different loading conditions. As a result of varying stress level, their transforamtion temepratures change and it may adversly affect the SMA-based actuators. Till date, there are no reports available to show the stress influence coefficient of NiTiCu based SMAs. This paper, therefore, aims to correlate quantitatively and qualitatively the influence of applied load/stress to the transition characteristics of an NiTiCu SMA subjected to SME cycling under constant stress.

2. Materials and Methods

In the present study, an $Ni_{50}Ti_{45}Cu_5$ (at. %) SMA wire with 100 mm length and 1.43 mm diameter was used. Thermomechanical cycling tests were conducted using a custom-built thermomechanical cyclic testing machine, where the two ends of the wire specimen were crimped and attached to the holder, which in turn is fastened to the electrical connection (Fig. 1). As the higher magnitude of stress decreases the fatigue life of an SMA component/device [28,29], it can be improved by reducing the level of load applied [19,30,31]. However, reducing the stress level decreases the recovery strain. In the present work, therefore, a minimum stress was chosen in such a way that the applied stress was sufficient to achieve a minimum of recovery strain of 2%, and it was found (based on the trial-and-error) that to achieve this level of recovery strain, a minimum applied stress of 25 MPa was needed. Therefore, a stress range of 25 MPa to 60 MPa was chosen, which was below the yield stress of the martensite phase, and the tests were conducted within this stress range. An electric current of 5 A (Joule heating) was allowed to pass through the wire for heating the sample, followed by cooling it artificially to room temperature using the cooling fans attached to the machine. A LASER extensometer and pyrometer were used to measure the displacement of the wire and temperature, respectively, during heating/cooling. One end of the sample was externally loaded by hanging a deadweight, based on the chosen stress level, while the other end was fixed. During cycling, the test data, such as temperature and strain, were measured using optical pyrometer and laser extensometer, respectively. These data were recorded and stored in the computer through an interface.

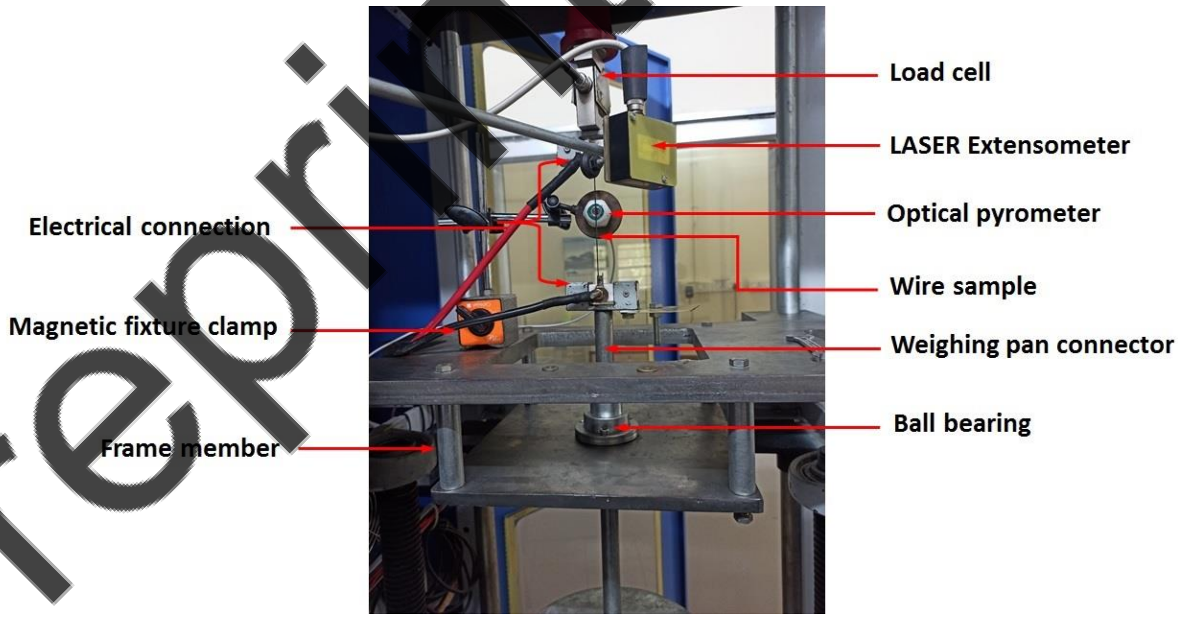

Fig. 1. Photograph of the thermomechanical cycling test setup

3. Results and Discussions

3.1 Stress influence coefficient

To understand the influence of applied load on recovery strain and transition temperatures, the strain vs. temperature curve at different applied stress levels for NiTiCu SMA was plotted. The phase transition temperatures were measured from the temperature vs. strain plot (Fig. 2) using the tangent method, and the recovery strain was calculated by taking the ratio of the length change to the original length (length in the martensitic phase after loading) and these values are given in Table 1.

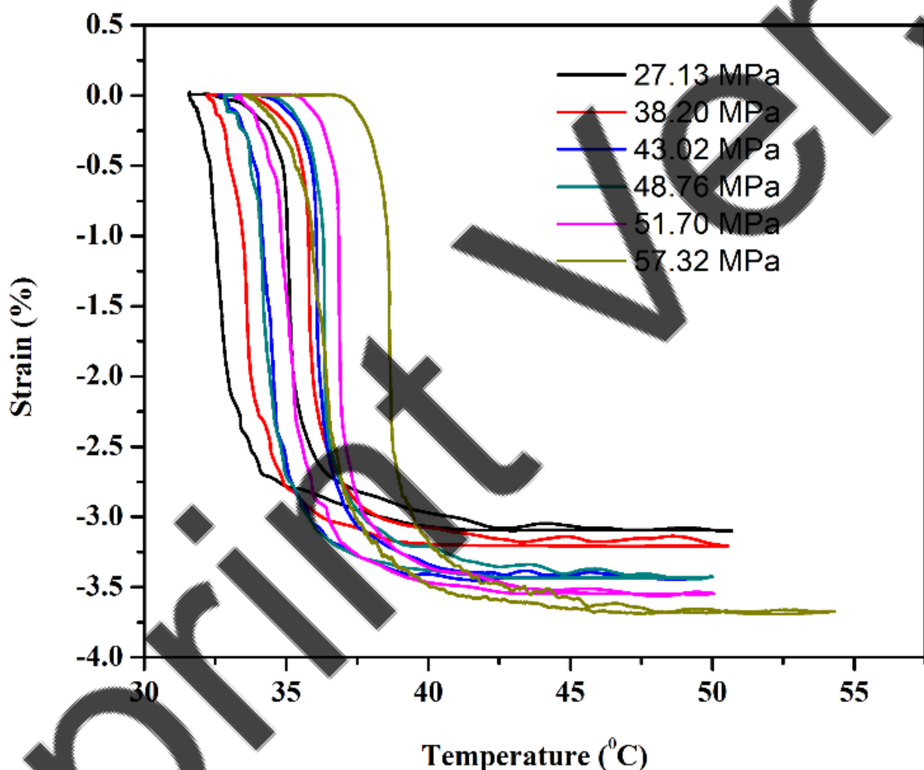

Fig.. 2. Strain vs. temperature plots at various stress levels for $Ni_{50}Ti_{45}Cu_5$ SMA wire

It can be observed from Fig. 2 that the transition temperatures are shifted to relatively higher temperatures with an increase in applied stress level. This is because of the stabilization of martensite brought about by the load applied during reverse transformation occurring at relatively higher temperatures than in the stress-free condition, as stated by the Clausius-Clapeyron relationship [12]. Moreover, the martensitic variants prefer to nucleate and grow in a direction that is parallel to the applied load due to the dislocations structure that developed during the thermomechanical cycling [18,32]. It is also evident that the extent of strain recovery also gradually increases with increasing applied load level, as shown in Fig. 3. Appling a stress of 57 MPa yields a maximum recovery strain of 3.7%. This is due to the fact that at a higher stress level, the extent of deformation is higher, and so is the recovery while heating it back to the austenitic phase.

Table 1. Effect of stress on phase transformation temperatures and recovery strain for $Ni_{50}Ti_{45}Cu_5$ SMA

Applied Stress [MPa]	M_f [°C]	M_s [°C]	A_s [°C]	A_f [°C]	Recovery strain (Max.) [%]
27.14	31.58	33.11	34.91	35.46	3.10
38.19	32.28	33.87	35.57	36.62	3.21
43.02	32.73	34.85	36.02	36.67	3.46
48.77	32.8	34.61	36.2	36.76	3.44
51.70	33.31	35.45	36.9	37.08	3.56
57.32	33.5	36.87	37.72	38.82	3.70

It is also observed that the hysteresis width remains unaffected by the load applied, and this observation is in agreement with the earlier results reported for $Ni_{50}Ti_{40}Cu_{10}$ shape memory alloy by D.C. Lagoudas [30]. This is due to the fact that the addition of Cu enhances the phase transformation compatibility by making the motion of austenite/martensite interface easier. Apart from the change in phase transformation temperatures, the recovery strain, also known as transformation strain, increases as well with increasing magnitude of stress, as shown in Fig. 3. It is attributed to the increase in deformation, which is recoverable during heating, with increasing stress.

The stress influence coefficient is one of the most important design parameters, which indicates the proclivity of the transition temperatures to the stress applied. It was determined from Fig. 3. The slope of the linear fit curve of each transformation temperature gives the SIC. Generally, a steeper line is an indication of stable transformation temperature on the application of stress. The stress influence coefficients for some of the Cu-, NiTi-based shape memory alloys are listed in Table 2**.**

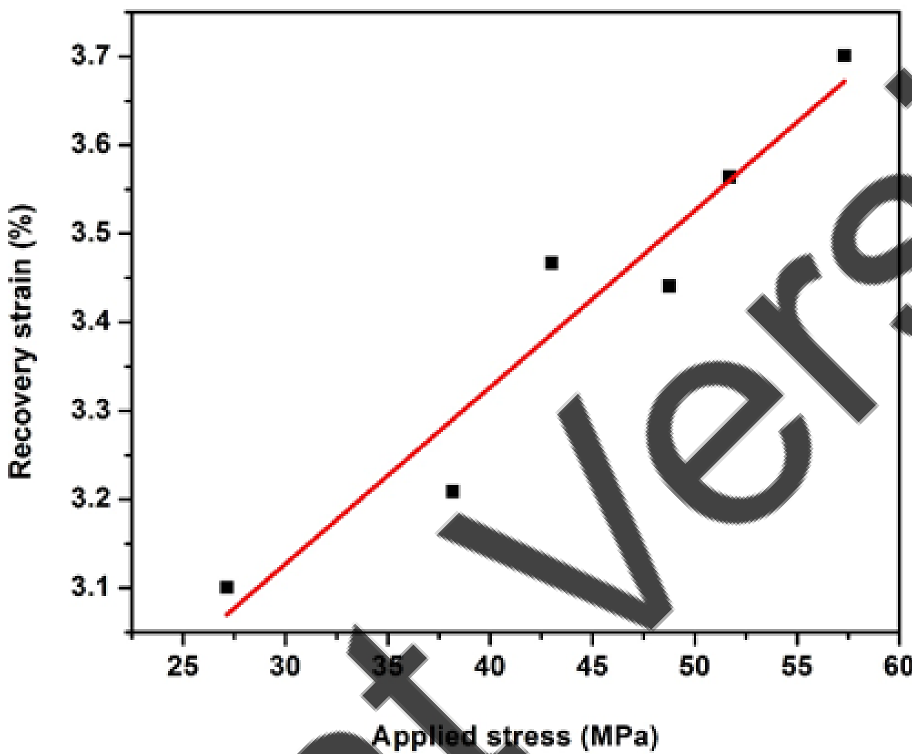

Fig. 3. Applied stress vs. maximum recovery strain plot of $Ni_{50}Ti_{45}Cu_5$ SMA at different magnitudes of stress

Table 2. Stress influence coefficients of some Ni-Ti and Cu-based SMAs as obtained from the literature and the present study

Sl.No.	Alloy system	Stress influence coefficient (MPa/℃)				Ref.
		(M_s)	(M_f)	(A_s)	(A_f)	
1.	$Ni_{50}Ti_{45}Cu_5$	7.74	15.13	10.46	8.82	Present study
2.	NiTi	2.2	3.6	7.2	4.2	[33]
3.	$Ti_{45.16}Ni$	38.1	-11	12.2	9.6	[34]
4.	CuZnAlCo	6.84	NR	7.24	NR	[35]

The stress influence coefficient values, which are listed here are based on the transformation temperatures and stress values reported in the respective literature [33–35], were found separately for each transformation temperature, i.e., M_s, M_f, A_s and A_f. In the literature [33,35], an average value of stress influence coefficient for martensite $(M_s+M_f)/2$, and for austenite $(A_s+A_f)/2$, respectively, are quoted. But from the present study, it is evident that the effect of stress on each of the transformation temperatures varies significantly. Hence, it is strongly recommended that the stress influence coefficient is reported separately for all transformation temperatures.

These results indicate that the phase transition temperatures of NiTiCu SMAs are influenced by the stress levels at different rates during thermomechanical cycling, as shown in Fig. 4. In the case of SE cycling, i.e., cycling at a constant temperature above A_f, the martensite that forms is stress-induced (SIM). But on the other hand, during SME cycling, i.e., cycling from above A_f to below M_f, both SIM (due to the applied stress) and thermoelastic martensite, TEM (due to cooling) form. The initial martensitic transformation (A to M) during SME cycling under constant stress is mainly due to the formation of SIM as the applied stress decreases the overall energy requirement. But the stress is insufficient to transform the whole volume of the parent phase into martensite as some of the variants may not be favorably oriented in the direction parallel to the applied stress. The variants that are not favorably oriented with the stress will transform into thermoelastic martensite, mainly due to the thermal energy contribution by cooling.

The stress influence coefficient of M_f is almost twice that for M_s, indicating that the martensite finish temperature is affected by the applied stress to a lesser extent. This behavior is attributed to the favorably-oriented martensite variants growing stronger in the presence of the applied stress (mostly influence the M_s), while those that are unfavorably-oriented (influence the M_f) are not much affected by the applied stress [36,37]. The martensite finish indicates the complete transformation of martensite from austenite. Therefore the M_f temperature does not depend much on the applied stress. Referring to the values presented in Table 2 and those obtained from the experiments from the present study, it can be well established that besides the applied stress, the alloying elements also exert an influence on the stress influence coefficient.

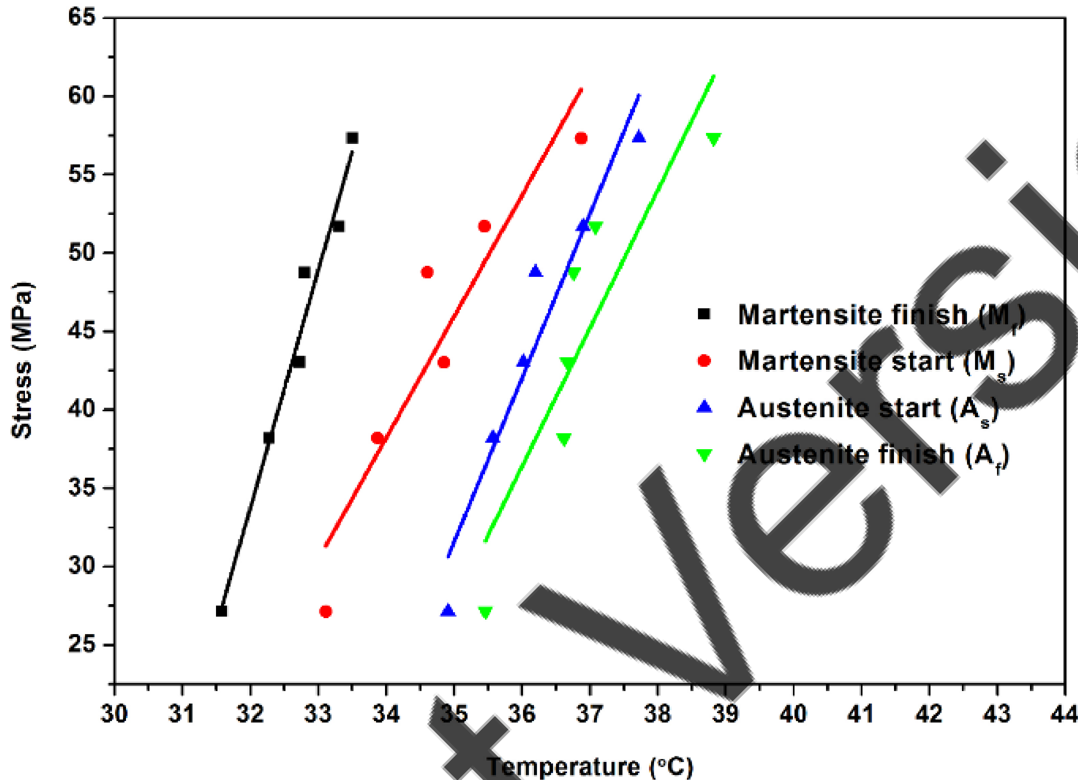

Fig. 4. Influence of stress applied on the transition temperatures

The stress influence coefficient values obtained from the present study clearly shows that the change in M_s (martensite start temperature) is more influenced by the applied stress rather than the other critical phase transformation temperatures. From Table 2, it can be seen that the stress influence coefficient varies with alloy composition. Generally, a higher value of SIC is an indication of highly stable transition temperatures with relevance to the stress applied.

3.2 Stress vs. actuation/retraction rate

Unlike conventional actuators, which require a separate sensor, a processor, and an actuator unit, those based on SMAs tend to replace them due to their high specific work output and simplicity of design [38,39]. SMA actuators actuate upon heating (M→ A transition) and retract upon cooling (A→ M transition). The rate of actuation and that of retraction varies depending on the operating conditions like rate of cooling, stress level, etc., which often lead to asymmetry of actuation and retraction. From Fig. 5, it is evident that stress has almost no effect on actuation during heating (M → A transition), while there is a significant change in retraction during cooling (austenite-to-martensite transformation). Moreover, symmetrical operations could be achieved at higher stress levels as compared to lower stresses. This is due to the fact that during heating, there is only one path that is energetically favorable for the reverse transition (M → A), irrespective of the orientation of the variants of martensite that is formed during cooling.

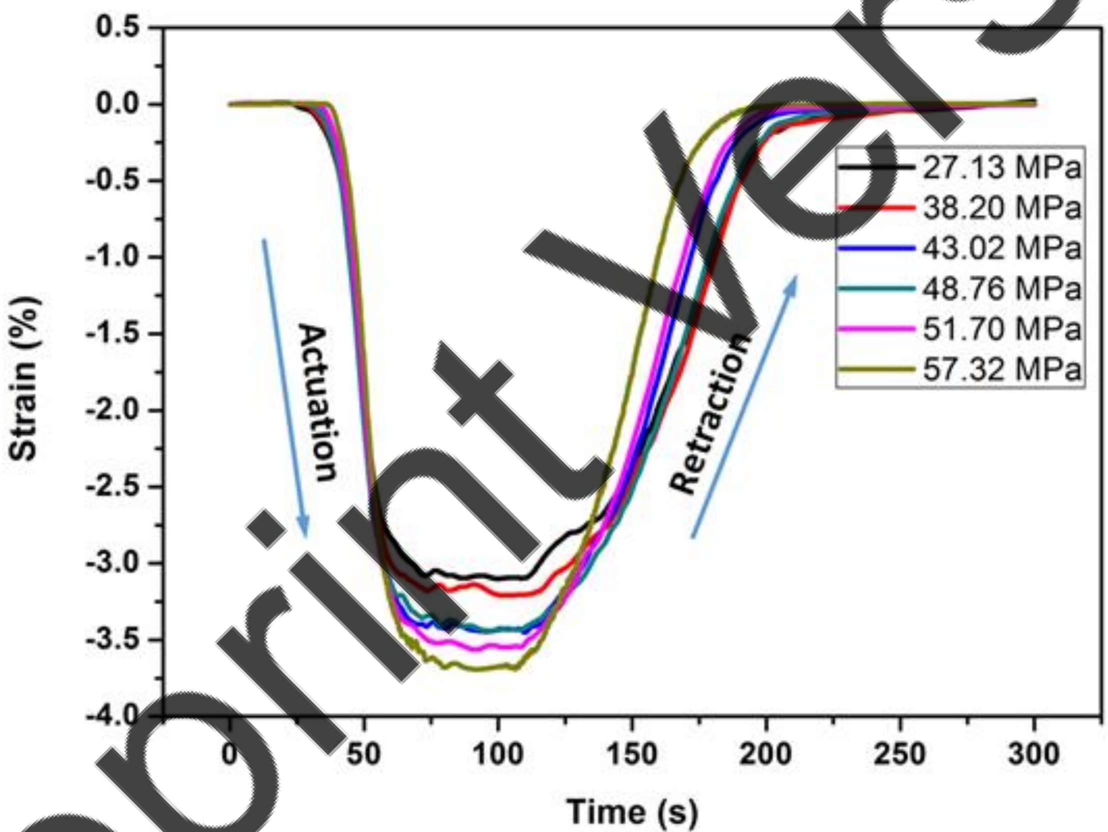

Fig. 5. Effect of applied stress on actuation/retraction time

Therefore, stress affects the rate of actuation only marginally. But, during cooling, the retraction starts earlier due to the rise in its M_s temperature with assistance from the applied stress, which facilitates the process of reorientation of martensite variants [40]. As a result, transformation progresses at a faster rate than the unstressed condition with higher symmetrical operation during actuation and retraction at higher rather than lower stress levels. The retraction time decreases (i.e., the increase in the rate of retraction) with the stress level being raised (Fig. 6). The reduction in the rate of retraction is not much significant at a lower value of stress, and the impact is greater at a higher magnitude of stress. These results indicate that the critical stress required for the effective reorientation of martensite variants is ~45 MPa, and after it reaches the critical value, the effect of stress is more prominent on the retraction rate. Another interesting observation from the present study is the recovery time required to increase the recovery strain from 1% to 2% is almost the same, while for 2% to 3%, it decreases with the stress level being increased (Fig. 6).

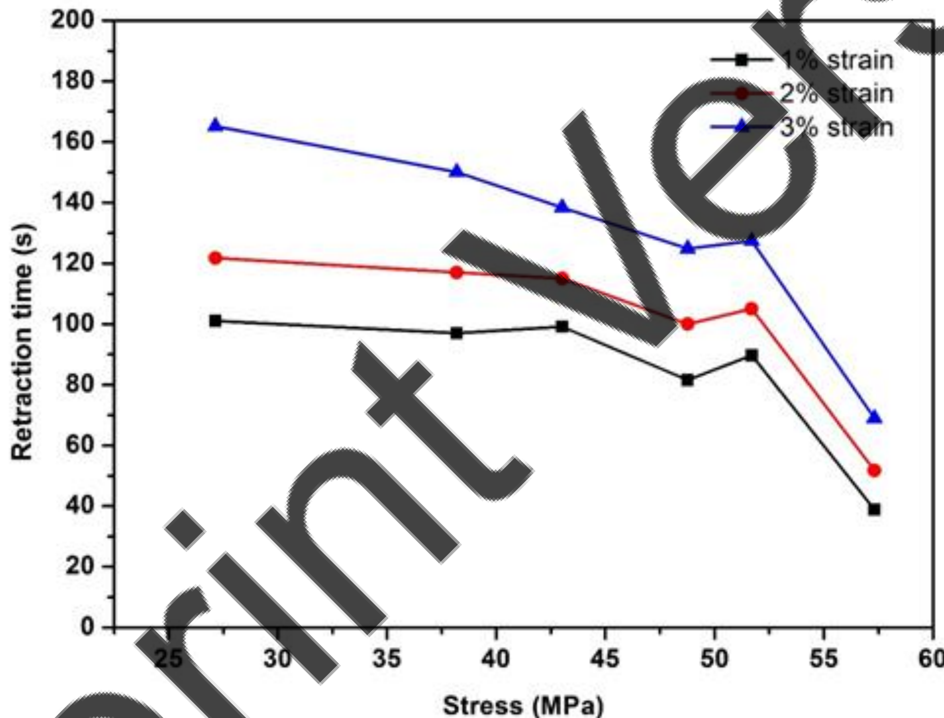

Fig. 6. Retraction time vs. stress plot for different strain levels

This is because the initial strain corresponds to the commencement of the martensitic transition, whereas the complete transformation corresponds to the final strain. As was discussed earlier, that the final transformation depends on the number of variants of martensite, which are not favorably aligned with the direction of applied stress, and such transformations depend only on the temperature. There is, therefore, a delay in the retraction at the final stages. But at higher stress levels, the orientation of the maximum number of martensite variants takes place. Along with the formation of thermoelastic martensite during cooling, stress-induced martensite by the applied stress facilitates the whole transformation to proceed faster and at a uniform rate. As a result, quicker retraction at higher stress levels.

Conclusions

In our study, the variation on SME characteristics (phase transition temperatures, actuation/retraction rate, hysteresis, recovery strain) of an $Ni_{50}Ti_{45}Cu_5$ (at.%) SMA wire subjected to constant stress during thermomechanical cycling was studied. The experimental study led to the following conclusions:

1. Applied stress affects the M_s temperature more significantly as compared to M_f, A_s, and A_f. This is because of the stabilization of martensite occurring at a relatively higher temperature.
2. An increase in applied stress level increases the recovery strain. The hysteresis width remains constant throughout the experiments, irrespective of the magnitude of stress applied.
3. The magnitude of stress applied has a larger impact on the retraction rate rather than the actuation rate and which makes the operation more symmetrical at higher stress levels.

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