

Functional fatigue in NiTi shape memory alloy wires - A comparative study

C.N. Saikrishna[#], K.V. Ramaiah^{#,*}, B. Vidyashankar^{a,b} and S.K. Bhaumik[#]

[#]Council of Scientific and Industrial Research (CSIR), National Aerospace Laboratories, Materials Science Division, Bangalore 560 017, INDIA, *Email: kvramaiah@nal.res.in

^aIndia Science Lab, General Motors Technical Center India Private Limited, ITPB, Bangalore 560 066, INDIA

^bPresently with GE John F Welch Technology Center, Bangalore 560 066, INDIA

ABSTRACT

The functional fatigue behaviour of two near equi-atomic NiTi shape memory alloy wires obtained from different sources were evaluated. Results showed that though the wires had similar transformation temperatures and mechanical properties, their functional fatigue behaviour upon thermo-mechanical cycling was at variance. Under a variable stress in the range 150-450 MPa and 4% recovery strain, one of the wires showed better stability, and significantly higher fatigue life (~30,000 cycles) than the other (~3,500 cycles). The reasons for such wide variation in thermo-mechanical fatigue behaviour have been discussed in this paper.

Keywords: Shape memory alloy; NiTi wire; Thermo-mechanical cycling; functional fatigue

1. INTRODUCTION

In recent times, near equi-atomic NiTi shape memory alloys (SMAs) have been used in a variety of engineering applications because of their superior shape memory behaviour, mechanical properties and corrosion resistance [1-2]. While the science of SMAs is known over past few decades, the processing of these alloys into product forms with consistent and repeatable properties has been a challenge till date. The properties of NiTi alloys are extremely sensitive to composition as well as thermo-mechanical processing [3]. An increase in Ni concentration by 0.1 at.% lowers the transformation temperatures by about 10°C [1,3]. Even for alloys having similar compositions, the properties vary significantly depending on processing history such as melting process adopted, percent cold work retained in the material, temperature/time of shape memory heat treatment, type of stabilization treatment, etc [4-8]. Studies have shown that NiTi alloys obtained from different sources and tested under similar conditions often have varied transformation and functional fatigue response [4, 9-14]. In this study, functional fatigue response of two NiTi wires obtained from different sources was evaluated upon thermo-mechanical cycling (TMC). The reasons for the variation in stress/strain response and fatigue life observed in these wires have been discussed.

2. EXPERIMENTAL PROCEDURE

Two near equi-atomic NiTi wires of diameter 0.37 mm obtained from different sources were used in this study.

While the first wire (wire-1) was fabricated in National Aerospace Laboratories, Bangalore, the second wire (wire-2) was obtained from a commercial source. The nominal composition of the wires was evaluated using wave length dispersive spectroscopy (WDS) attached to Cameca make electron probe micro analyzer (EPMA, Model: SX 100). The transformation temperatures were determined using a TA make differential scanning calorimeter (DSC, Model: Q2000). The DSC scans were carried out at a heating/cooling rate of 10°C/min under nitrogen atmosphere. The tensile properties of the wires were evaluated using Instron make table top universal testing machine (UTM) of 5 kN capacity. Wire samples of gauge length 30 mm were tested as per ASTM E8M standard at strain rate of 2×10^{-3} /s. Stress rate tests were carried out to determine the rate of recovery stress generated in the wires per degree rise in temperature. The test was carried out by pre-straining the wire to 4% and then heating it in the constrained condition. The stress generated in the wires for every 10°C rise in temperature was recorded. TMC tests were carried out on wire sample of gauge length 150 mm under a variable stress in the range 100-450 MPa and 4 % recovery strain using a bias spring. The details of the test set-up used can be found in ref. [7]. During TMC, the wires were heated by resistive heating and the cooling was effected by natural air cooling. The current and time schedule adopted for the tests is shown in Fig. 1. The thermal cycling schedule was so chosen that the maximum stress generated in the wires in high temperature austenite phase was 450 MPa. The wires were tested till fracture and the variation in stress/strain with number of cycles were recorded. The two way shape memory strain (TWSMS) was determined by subjecting the

wires to TMC at a constant stress of 50 MPa. The difference in length of the wires in martensite and austenite phases determined from the transformation curve, that is, strain vs. temperature curve at this stress has been reported as the TWSMS. The variation in austenite transformation temperatures under stress were determined using similar transformation curves generated from TMC tests at different stress levels ranging from 50-600 MPa.

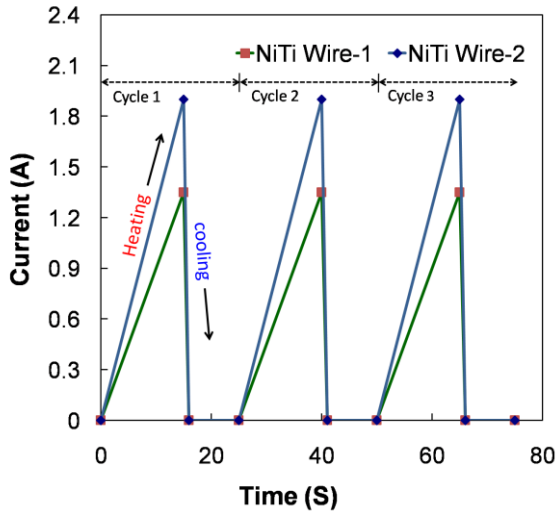


Figure 1 Current vs. time schedule used for heating and cooling of NiTi wires during TMC

3. RESULTS

Compositional analysis through EPMA confirmed that both the wires were fabricated from 49.8Ni-50.2Ti (at.%) alloy.

The DSC scans of the wires under stress-free condition are shown in Fig. 2. The wires have more or less similar transformation behaviour except for the diffused nature of transformation in wire-1. An occurrence of R-phase is observed in these wires prior to the martensitic transformation on cooling. The transformation temperatures of the wires are within $\pm 2^\circ\text{C}$ except for the martensite finish temperature, which are 19 and 25°C for wire-1 and wire-2 respectively (Fig. 2 inset). The transformation hysteresis in both the wires is $\sim 30^\circ\text{C}$.

The stress vs. strain plot of the NiTi wires tested in martensite phase at room temperature (23°C) is shown in Fig 3. Both the wires show similar tensile strength (1390-1450 MPa) and percent elongation to failure (10.4-11.4%). The martensitic stress plateau in the plot is not discernible as there is a monotonous increase in stress/strain values with applied stress.

Figure 4 shows the plot of recovery stress generated in the wires in constrained condition as a function of temperature. The rate of recovery stress generation, stress rate, in the wires calculated from the slope of the curve in Fig. 4 is similar, $7.4 \text{ MPa}/^\circ\text{C}$ for wire-1 and $6.9 \text{ MPa}/^\circ\text{C}$ for wire-2. However, at a particular temperature, the recovery stress generated in wire-1 is 70-100 MPa higher than that generated in wire-2. For example, at 115°C , the recovery stress generated in wire-1 is 450 MPa as compared to 365 MPa in wire-2. In other words, to attain a recovery stress of 450 MPa, wire-2 needs to be heated to a temperature of 130°C , 15°C more than that required for wire-1 (Fig. 1 and 4).

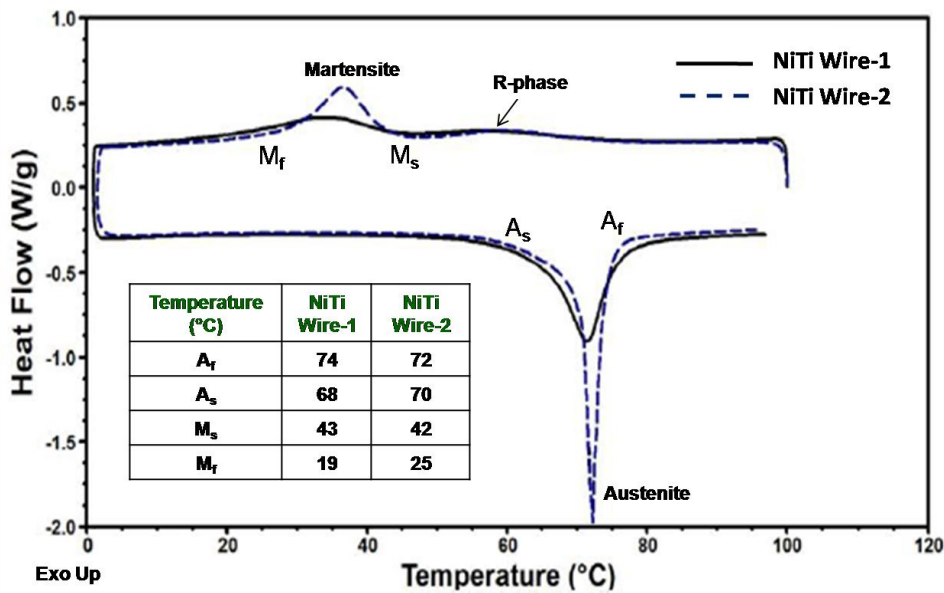


Figure 2 DSC scans shows the transformation behaviour of NiTi wires

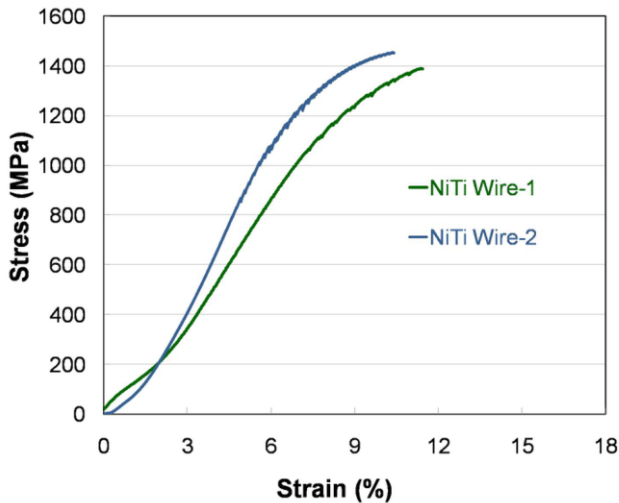


Figure 3 Stress vs. strain plot of NiTi wires in martensite phase

The strain vs. temperature plot of wires upon TMC at a constant stress of 50 MPa is shown in Fig. 5. It can be seen that at room temperature (23°C), wire-1 has a considerably lower TWSMS of ~3.0% compared to that of ~4.5% in wire-2. Also, though the forward transformation start temperature (M_s) for the wires is within 1-2°C, significant under-cooling is required in case of wire-1 for completion of transformation. The martensite finish temperature (M_f) in wire-1 and wire-2 at 50 MPa stress was determined to be 20°C and 30°C respectively. The transformation hysteresis indicated by the width of the transformation curve is of similar magnitude in both the wires. However, the slope of the transformation curves is significantly different such that wire-1 has a higher tilt towards the left. The variation in

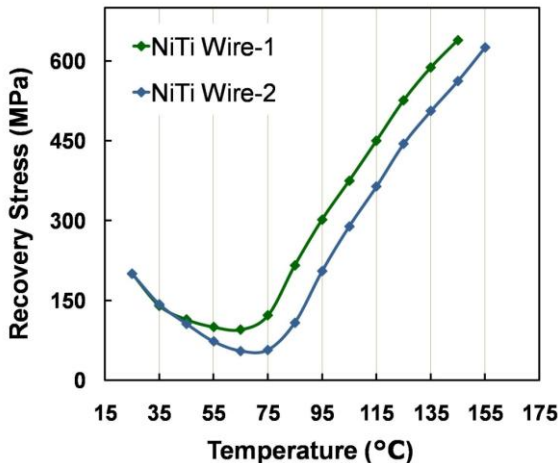


Figure 4 Stress vs. temperature plot shows the recovery stress generated after 4% pre-strain and heating in constrained condition

transformation temperatures (A_s : austenite start, A_f : austenite finish) under load determined through TMC at different stress levels ranging from 50-600 MPa is shown in Fig. 6. It can be seen that the slope of A_s and A_f as a function of stress is higher in wire-1 than that in wire-2. This indicates less stress dependency of transformation temperatures in wire-1. Hence, to achieve a similar recovery stress, wire-1 requires heating to lower temperature than that of wire-2. It may be noted that slope of curve in Fig. 6 represents the Clausius-Clayperon relationship for the wires.

The plot of stress/strain vs. No. of cycles of TMC under a variable stress in the range 150-450 MPa and 4% recovery strain is given in Fig. 7. It can be seen that wire-1 and wire-2 display wide variation in fatigue response upon TMC. Wire-1 shows better stability in respect of stress/strain variation with increase in number of cycles as compared to that of wire-2. While the stress levels in wire-1 is largely maintained in both austenite and martensite phase till fracture, there is substantial decrease in stress levels in wire-2. Strain vs. No. of cycles plot also show a similar variation. Wire-1 shows a relatively stable strain response till fracture as compared to significant decrease in recovery strain (RS) and increase in remnant deformation (RD) in wire-2. Also, wire-1 exhibited significantly higher fatigue life (~30,000 cycles) than that of wire-2 (~3,500 cycles).

4. DISCUSSION

The transformation and functional fatigue properties of the NiTi SMAs are extremely sensitive to chemical composition and the subsequent thermo-mechanical processing history. The wires evaluated in the present study have similar chemical composition and therefore, the variations observed in their fatigue response upon TMC can be attributed to

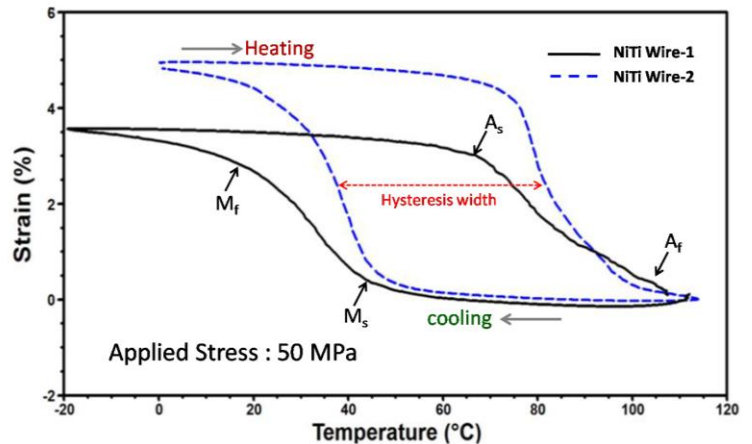


Figure 5 TWSMS vs. temperature of NiTi Wire-1 and Wire-2 upon TMC

differences in their thermo-mechanical processing history.

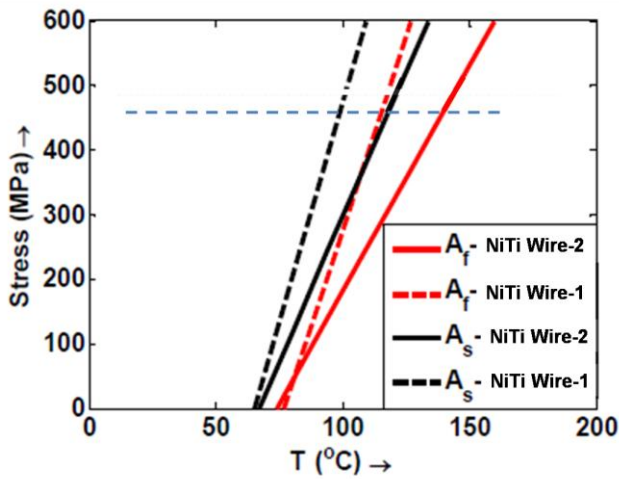


Figure 6 Stress vs. temperature plot of NiTi wires indicating austenite temperatures

Results show that the NiTi wires obtained from different sources have similar transformation temperatures and mechanical properties (Fig. 2 and 3). However, variation in the properties of wires was observed with regard to M_f and the maximum wire temperature in austenite phase for achieving a predetermined recovery stress level (Fig. 2 and 4). From the stress vs. strain plot, it can be seen that the martensitic stress plateau is not discernable in both the wires (Fig. 3). This is because of high TWSMS in the wires at room temperature (Fig. 5). It is well known [15-17] that the origin of TWSMS in SMAs is residual internal stresses and the presence of preferably oriented martensite variants in the

microstructure. The combination of these two causes detwinning in martensite without the application of any external stress. The residual stresses in SMA wires are generally a result of either thermo-mechanical processing and/or post processing stabilization treatment. Considering the magnitude of TWSMS (Fig. 5), it is apparent that the wires have differing residual stresses as well as orientation of martensite variants. This, in turn, indicates that there are subtle differences in the processing of the two wires, which are not reflected in gross thermo-physical properties or quasi-static mechanical/functional properties.

The stress-free transformation temperatures of the wires used in the study are within $\pm 2^\circ\text{C}$, except for M_f temperature which is significantly lower in wire-1 (Fig. 2). The change in free energy during phase transformation essentially consists of chemical and non-chemical energies. The non-chemical energy has reversible and irreversible components. The reversible component in this, is due to the elastic strain energy stored in the material as a result of formation of large number of martensite twin variants. The formation of martensite twins is associated with the accommodation of Bain strain resulting from the change in crystal structure during phase transformation. Under external stress, detwinning of martensite takes place with variants favourably oriented in the stress direction and the volume fraction of detwinned martensite grows at the expense of those less favourably oriented. In this process, the stored elastic strain energy is released. The irreversible component in the non-chemical energy is mainly due to the dissipation of frictional energy associated with the movement of interfaces during phase transformation and its interaction with the defects/precipitates in the microstructure [18]. The strain energy stored in the system aids the reverse transformation, that is, martensite to austenite phase. Hence,

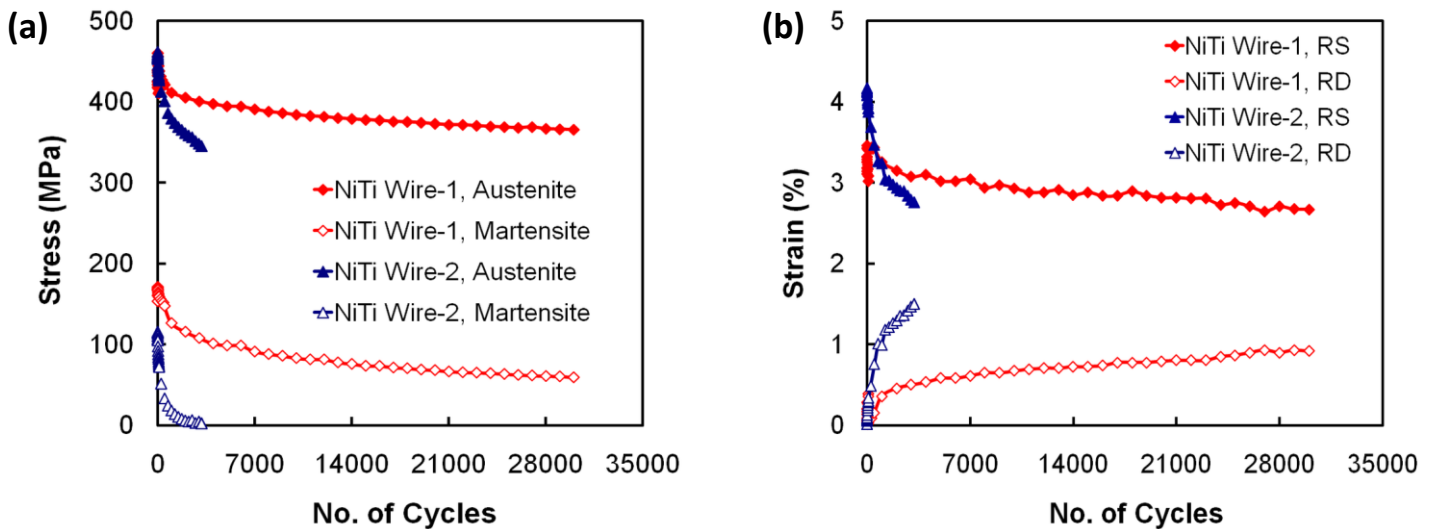


Figure 7 Thermo-mechanical cycling behaviour of NiTi wires under variable stress (a) Stress vs. No. of cycles, (b) Strain vs. No. of cycles (RS: Recovery strain, RD: Remnant Deformation)

less energy in terms of heat input is required to complete the transformation. On the other hand, during forward transformation, austenite to martensite phase, more undercooling is necessary as the stored elastic strain energy opposes the transformation [18]. The lower M_f temperature observed in wire-1 indicates that the stored elastic energy in this case is higher than that in wire-2 (Fig. 2). The tilt in transformation curve towards left (Fig.5) also substantiates the high stored elastic energy in wire-1 [19]. The role of stored elastic strain energy on recovery stress can be understood from Fig. 4, wherein, at a particular temperature, the recovery stress generated in wire-1 is higher than that in wire-2. This is because, the transformation from martensite to austenite phase under stress is assisted by the stored elastic strain energy in the material and hence, the transformation takes place at lower temperatures. Therefore, during reverse transformation, at any given temperature, wire-1 has more volume fraction of transformed austenite compared to that in wire-2, resulting in higher recovery stress. This is clearly evident from the stress dependency of austenite transformation temperatures of the wires shown in Fig. 6.

As discussed above, under a particular set of TMC conditions, that is, stress range and recoverable strain, wire-2 required heating to a higher temperature than that of wire-1. It is well known [6,20] that fatigue life of SMA wires upon TMC decreases drastically as the temperature in the austenite phase increases. Hence, the wide variation observed in fatigue life of the SMA wires, viz., 30,000 cycles for wire-1 and 3,500 cycles for wire-2 can be attributed to this aspect (Fig. 7). Also, the propensity to generate defects in the microstructure gets accelerated as the maximum temperature of the SMA wire increases during TMC [6,20-21]. Therefore, the higher RD observed in wire-2 is justifiable. The RD in the SMA is a measure of untransformed austenite phase during thermal cycling. Therefore, as RD increases, less and less volume percent of martensite phase is available for contributing to transformation strain in the material. As a result, RS and stress generated in wire-2 were significantly lower than that in wire-1.

5. CONCLUSIONS

A comparative study on the functional fatigue behaviour of two near equi-atomic NiTi shape memory alloy wires obtained from two different sources was undertaken. Based on the experimental results, the following conclusions can be drawn.

- In spite of having similar composition, transformation temperatures and mechanical properties, the wires have shown wide variations in the functional fatigue behaviour. This variation in functional fatigue behaviour is found to be because of the difference in maximum

temperature attained in the wires during TMC. Under a variable stress in the range 150-450 MPa and 4% recovery strain, wire-1 showed better stability in respect of stress/strain response, and significantly higher fatigue life of ~30,000 cycles compared to ~3,500 cycles in wire-2.

- Thermomechanical processing history of SMA wires has a significant bearing on its functional fatigue behaviour. The variation in stored elastic strain energy in the material originating from the processing history and the post processing stabilization treatment has significant effects on the functional fatigue behaviour of NiTi SMA wires.

ACKNOWLEDGEMENTS

The work presented in this study was carried out under the XI 5-Year Plan Project of CSIR-NAL, India. The authors acknowledge the contributions of D. Paul in conducting the experiments. The authors thank Director, NAL for permission to publish this work.

REFERENCES

- [1] Humbeeck, J.V., "Shape memory alloys: A material and a technology", *Adv. Eng. Mater.*, 3, 837-850 (2001)
- [2] Butera, F., "Shape memory actuators", *Adv. Mater. and Processes*, 37- 40 (2008).
- [3] Pelton, A.R., Russel, S.M., DiCello, J., "The physical metallurgy for Nitinol for medical applications", *JOM*, 55, 33-37(2003)
- [4] Patel, M.M., Plumley, D.L., Bouthot, R.J., Proft, J.L., "The significance of melt practice on fatigue properties of superelastic NiTi fine diameter wire", *Proc. SMST-2006, USA, ASM International*, 43-52 (2008).
- [5] Morgan, N., Wick, A., DeCello, J., Graham, R., "Carbon and Oxygen levels in Nitinol alloys and the implications for medical device manufacture and durability", *Proc. SMST-2006, USA, ASM International*, 821-828 (2008).
- [6] Bertacchini, O.W., Lagoudas, D.C., Patoor, E., *Smart Structures and Materials 2003: Active Materials: Behavior and Mechanics*, (ed.) D.C. Lagoudas, *Proc. of SPIE* 5053, 612-624 (2003).
- [7] Saikrishna, C.N., Ramaiah, K.V., Allam Prabhu, S., Bhaumik, S.K., "On the stability of NiTi wire during thermo-mechanical cycling", *Bull. Mater. Sci*, 32, 343-352 (2009)
- [8] Malard, B., Pilch, J., Sittner, P., Delville, R., Curfs, C., "In situ investigation of the fast microstructure

- evolution during electropulse treatment of cold drawn NiTi wires”, *Acta Materialia* 59, 1542-1556 (2011)
- [9] Marquez, J., Slater, T., Sczerzenie, F., “Determining the transformation temperatures of NiTi alloys using differential scanning calorimetry”, SMST-1997, California, USA, 13-18 (1997).
- [10] Tsoi, K.A., Schrooten, J., Stalmans, R., “Part I. Thermomechanical Characteristics of shape memory alloys”, *Mater. Sci. Eng. A*, 368, 286-298 (2004).
- [11] Duda, S.H., Wiskirchen, J., Tepe, G., Bitzer, M., Kaulich, T.W., D. Stoeckel, C.D. Claussen, “Physical Properties of Endovascular Stents: An Experimental Comparison”, *JVIR*, 11, 645-654 (2000)
- [12] Henderson, E., Nash, D.H., Dempster, W.M., “On the experimental testing of fine nitinol wires for medical devices”, *J. Mech. Behav. Biomed*, 4, 261-268 (2011)
- [13] Johnson, E., Lloyd, A., Kuttler, S., Namerow, K., “Comparison between a Novel Nickel-Titanium Alloy and 508 Nitinol on the Cyclic Fatigue Life of ProFile 25/.04 Rotary Instruments”, *J. Endod*, 34, 1406-1409 (2008)
- [14] Schaffer, J.E., “Structure-property relationships in conventional and nano-crystalline NiTi intermetallic alloy wire”, *J. Mater. Eng. Perform*, 18, 582-587 (2009)
- [15] Perkins, J., Edwards, G.R., Such, C.R., Johnson, J.M., Allen, R.R., [Shape Memory Effects in Alloys], Plenum Press, New York, 273-303 (1975)
- [16] Liu, Y., McCormick, P.G., “Factors influencing the development of two way shape memory in NiTi”, *Acta Mater.*, 38, 1321-1326 (1990).
- [17] Perkins, J., Sponholz, R.O., “Stress-Induced Martensitic Transformation Cycling and Two-Way Shape Memory Training in Cu-Zn-Al Alloys”, *Metall. Trans. A* 15, 313-321 (1984).
- [18] Pulumbo, M., “Thermodynamics of martensitic transformations in the framework of the CALPHAD approach”, *CALPHAD*, 32, 693-708 (2008)
- [19] Humbeeck, J.V., Stalman, R., Chandrasekaran, M., Delaey, L., [Engineering aspects of shape memory alloys], T.W. Duerig et al., eds., Butterworth-Heinemann, 96-105 (1990)
- [20] Bhaumik, S.K., Saikrishna, C.N., Ramaiah, K.V., “Characteristic Behaviour of NiTi SMA Wire undergoing Thermo-mechanical Cyclic Loading”, *Proc. ISSS 2012, IISc, Bangalore, India, 2012.*
- [21] Hornbogen, E., “Thermo-mechanical fatigue of shape memory alloys”, *J. Mater. Sci.*, 39, 385-399 (2004).