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On stability of NiTi wire during thermo-mechanical cycling

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Abstract. **The use of NiTi wire as thermal actuator involves repeated thermal cycling through the transformation range under a constant or fluctuating load. The stability of the material under such conditions has been a concern for the past many years. Experimental results show that for a given alloy composition, the repetitive functional behaviour of NiTi wire is largely dependent on the processing schedule/parameters and the stress–strain regime of thermo-mechanical cycling (TMC). Among the various processing parameters, retained cold work in the material and the shape memory annealing temperature/time have significant influence. It has been shown in the present study that for a stable functional behaviour, the material needs to be tailored through judicious selection of these parameters. Study also shows that, after processing, the material requires an additional stabilization treatment for ensuring minimal variation in the repetitive functional response upon TMC.**

Keywords. **Shape memory alloy; NiTi; wire; processing; mechanical and functional properties; stability.**

1. Introduction

Near equi-atomic NiTi shape memory alloys (SMAs) are potential candidates for functional applications such as actuators in aerospace, engineering and biomedical systems (Humbeeck *et al* 1990). Binary NiTi SMAs have the advantage of superior shape memory properties viz. recoverable strain and recovery stress, better thermal and phase stability, and higher fatigue life compared to those in other SMA systems (Humbeeck *et al* 1990; Melton 1990; Saburi 1998). The properties of NiTi SMAs are very sensitive to alloy chemistry and thermo-mechanical processing. The alloy composition largely determines the overall property range for the NiTi alloy. However, the properties of the alloy can be further tailored to a great extent by judicious selection of processing parameters such as retained cold work and shape memory annealing temperature/time.

During thermal actuation, the SMA wire undergoes repeated temperature cycles through the transformation range under constant/variable load, referred to as thermo-mechanical cycling (TMC). Studies (Perkin and Sponholz 1984; Stachowiak and McCormick 1988; Humbeeck 1991; Eggeler *et al* 2004; Saikrishna *et al* 2006; Ramaiah *et al* 2008; Bhaumik *et al* 2008) have shown that the functional properties of the SMA such as the transformation temperatures, transformation hysteresis, and strain response etc change continuously upon TMC. This unstable behaviour is attributed to the generation of defects in the microstructure during TMC (Perkin and Sponholz 1984; Stalmans *et al* 1991; Jiang *et al* 1997; Liu *et al* 1999; Bhagyaraj *et al* 2009). The response of the NiTi SMA upon TMC largely depends on the thermo-mechanical processing history and the stress–strain regime of TMC, and is directly related to the defects density in the material (Erbstoeszer *et al* 2000; Bhaumik *et al* 2008; Ramaiah *et al* 2008).

The present study deals with the effect of retained cold work and the shape memory annealing temperature on the stability of NiTi wire during TMC.

2. Experimental

NiTi wire specimens of 0·5 mm ϕ having a nominal composition of 49·8Ni50·2Ti (at.%) were used for the present study. Non-consumable vacuum arc melting process was used for alloy preparation. Re-melting was carried out six times to get a homogeneous melt. The wires with varying retained cold work in the range 20–40% were obtained by subjecting the cast alloy to standard metal forming operations such as hot rolling, cold rolling and cold wire drawing. The as-drawn wires were shape memory annealed at temperatures in the range 350–500°C for 15 min followed by water quenching (table 1).

The tensile properties of the wire samples in martensite and austenite phase were evaluated using universal testing machine (5 kN capacity, Instron make) as per ASTM-E8M standard. The tests were carried out on specimens of gauge length 40 mm at a strain rate of $2{\cdot}5 \times 10^{-3}$ s^{-1}.

The TMC experiments on the wire specimens were carried out at a stress of 200 MPa with no constraint on the deformation and strain recovery. The test set-up used

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Table 1. Properties of NiTi wires processed under varying conditions (CW: cold work; M: martensite phase; A: austenite phase).

	Processing parameters		UTS (MPa)		Elongation to fracture (%)		Plateau strain (%)	
Sample	CW (%)	Annealing temp. (°C)*	*M*	*A*	*M*	*A*	*M*	*A*
S1	20		1227	1134	27	32	–	–
S2	30	350	1377	1330	17	16	–	6·9
S3	40		1555	1577	16	19	–	6·3
S4	20		1171	1118	23	29	4·7	8·2
S5	30	400	1300	1225	15	19	4·8	7·8
S6	40		1510	1497	18	16	4·8	7·5
S7	20		1146	1038	33	41	5·3	9·9
S8	30	450	1199	1089	15	18	5·0	8·5
S9	40		1234	1125	16	17	5·7	9·8
S10	20		1084	1033	28	35	5·6	10·4
S11	30	500	1112	1003	62	58	5·2	9·8
S12	40		1080	994	67	54	5·5	10·1

*Annealing time kept constant at 15 min

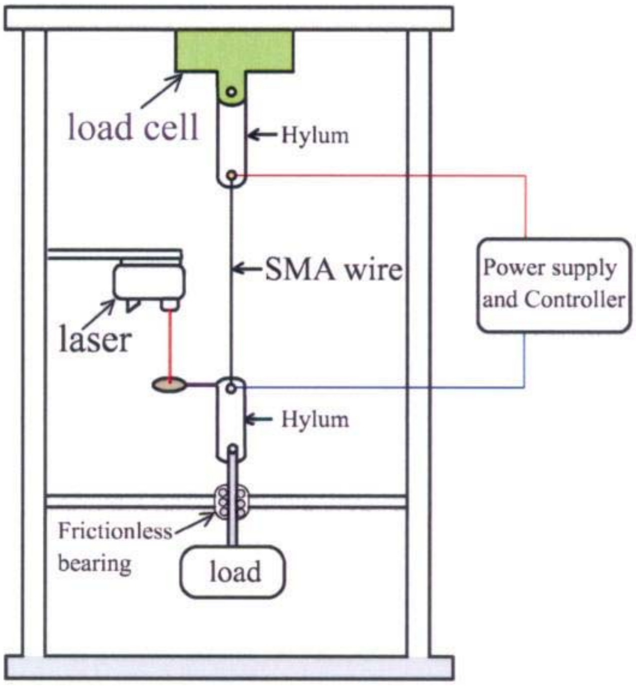

Figure 1. Schematic of the set-up used for thermo-mechanical cycling experiments.

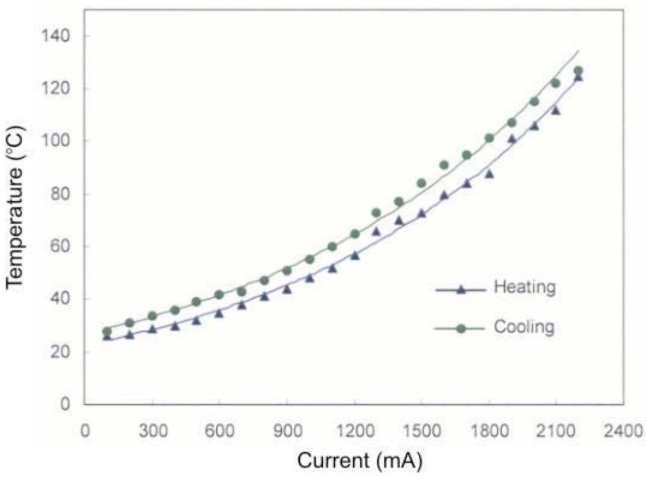

Figure 2. Current vs temperature plot for NiTi wire of 0·5 mm diameter.

is shown in figure 1. Resistive heating and forced air-cooling with a cycle time of 30 s each was used for the experiments. The temperature range for TMC was fixed in the range 25–100°C. A d.c. current of 2 A was required to heat the wire to ~ 100°C (figure 2). The length of the wire specimens used for each test was 280 mm. The displacement of the wire during TMC was monitored using a non-contact laser device of resolution, 10 μm. The recovery strain (RS) and remnant deformation (RD) were recorded continuously throughout the experiment. The transformation hysteresis loop (strain vs current) was recorded after predetermined number of cycles at a relatively slower heating/cooling rate.

A set of experiments was also carried out wherein the wire specimens were initially subjected to TMC at a higher stress of 300 MPa for 50 cycles and then, the TMC was continued at a stress of 200 MPa for 500 cycles.

3. Results

3.1 *Stress–strain behaviour*

3.1a *Effect of retained cold work and shape memory annealing*: The tensile properties of NiTi wire specimens in martensite and austenite phases with varied retained cold work and shape memory annealing temperature are given in table 1 and figures 3–5. Examination of

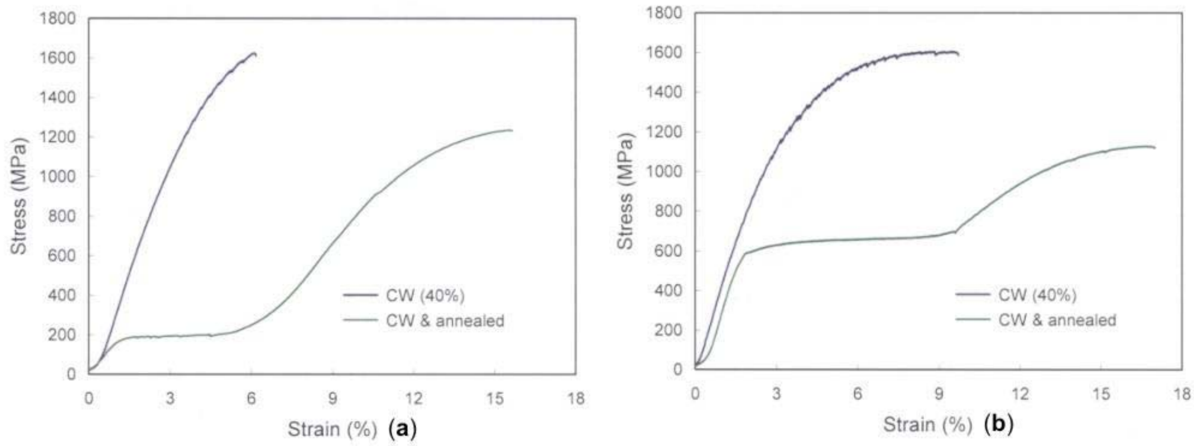

Figure 3. Stress vs strain plots of NiTi wires with 40% cold work before annealing and after annealing at 450°C for 15 min: (**a**) martensite phase and (**b**) austenite phase.

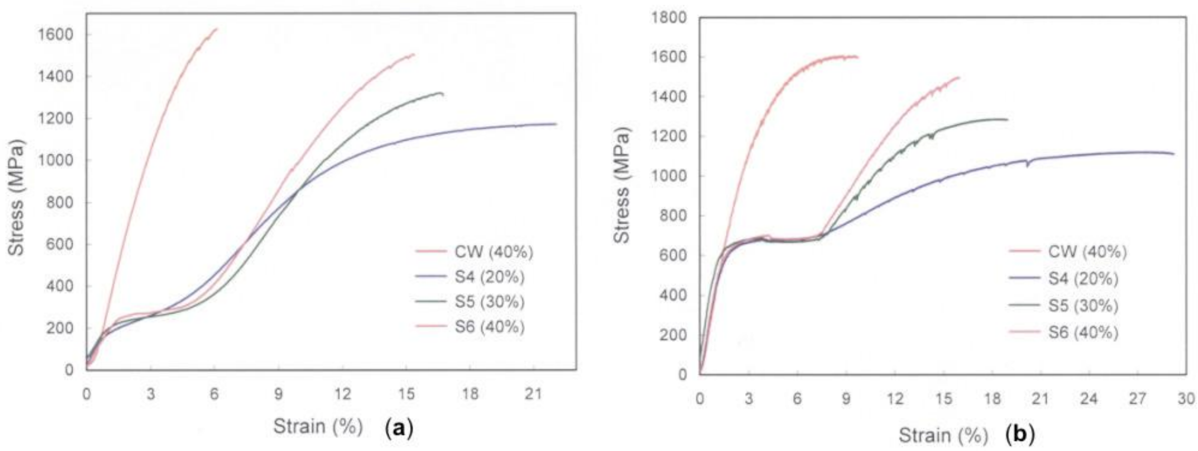

Figure 4. Stress vs strain plots of NiTi wires with varying cold work and after annealing at 400°C: (**a**) martensite phase, and (**b**) austenite phase.

the results indicate the following salient features: (i) the stress–strain behaviour of cold worked NiTi wire is similar to that of any engineering material and it deviates from this behaviour only when the cold worked material is subjected to shape memory annealing treatment (figure 3), (ii) the yield strength and the ultimate tensile strength (UTS) increase with increase in retained cold work for wires annealed at temperatures up to 450°C (figure 4). For the wires annealed at 500°C, which is close to the recrystallization temperature range for the alloy, the variation in yield strength and UTS with cold work is marginal, (iii) percent elongation decreases with the increase in cold work from 20–30% at all annealing temperatures up to 450°C. Also, within this annealing temperature range, the variation in percent elongation to failure for the wires with cold work 30–40% is marginal and is in the range 15–19% (table 1 and figure 5). The percent elongation increases substantially when annealing is carried out at 500°C, (iv) for a given amount of cold work, the UTS decreases with increase in annealing temperature (table 1) and (v) plateau strain in SMAs is generally associated with the deformation in the material resulting from detwinning in martensite phase or stress induced martensite (SIM) transformation in the austenite phase. It can be seen that in general, the plateau strain in martensite phase is more or less the same with variation in percent cold work and annealing temperature. The plateau strain in austenite phase on the other hand, increases with increase in annealing temperature (table 1).

3.1b *Effect of thermo-mechanical cycling*: The effect of TMC on the stress–strain behaviour of NiTi wire is found to be quite significant. The yield strength increases with the increase in number of cycles of TMC (figure 6).

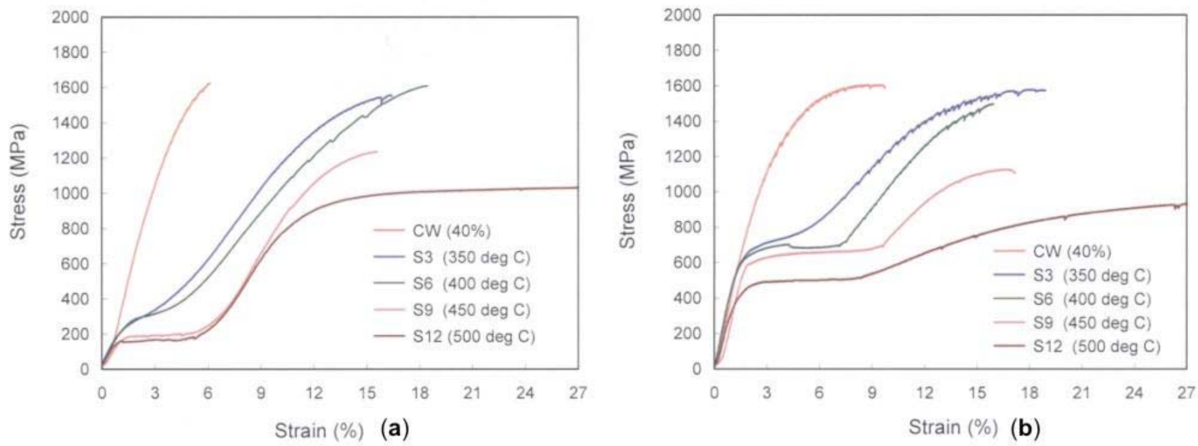

Figure 5. Stress vs strain plots of NiTi wires with 40% cold work as a function of annealing temperature: (**a**) martensite phase and (**b**) austenite phase.

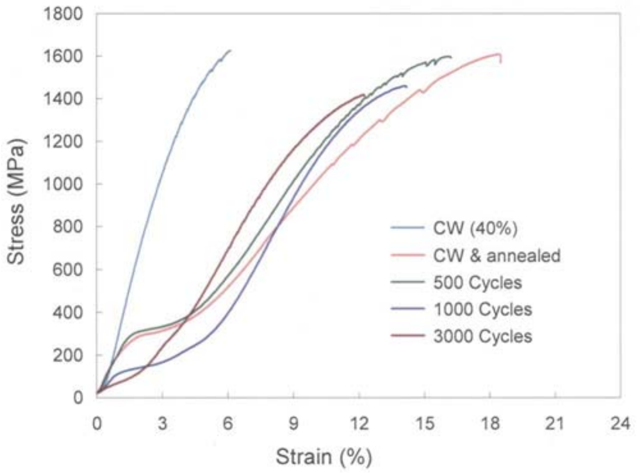

Figure 6. Stress–strain behaviour of NiTi wires with 40% cold work and annealed at 400°C subjected to TMC at 200 MPa.

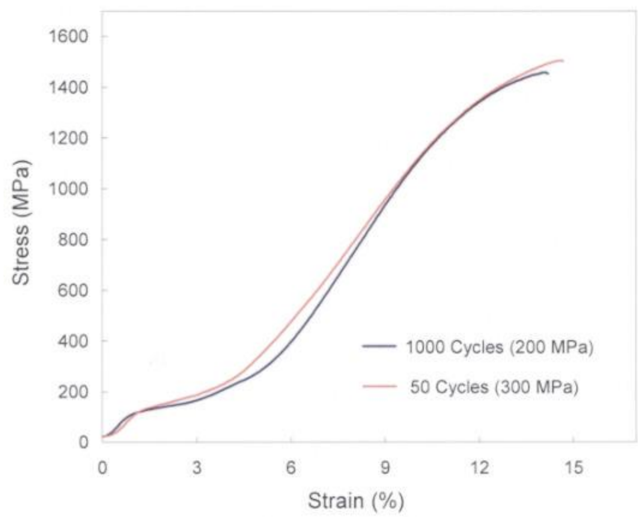

Figure 7. Stress–strain behaviour of NiTi wires with 40% cold work and annealed at 400°C after TMC at 200 MPa for 1000 cycles and 300 MPa for 50 cycles.

The other discernible change observed in the tensile properties was the decrease in the percent elongation. As TMC progresses, the nature of stress–strain curve tends to shift towards that of the cold worked material. The rate at which the stress–strain behaviour changes has a strong dependence on the stress at which the TMC is carried out, being higher at higher stress levels. In the present case, it has been observed that the stress–strain behaviour of the NiTi wire upon TMC at 300 MPa for 50 cycles is similar to that of the wire cycled at 200 MPa for 1000 cycles (figure 7).

3.2 *Functional behaviour upon TMC*

3.2a *Recovery strain (RS) and residual deformation (RD)*: The strain response of the NiTi wire upon TMC is presented in this section. In the description, 'RS' refers to the strain recovered upon heating (martensite → austenite phase transformation) during the *n*th cycle while 'RD' refers to the plastic strain accumulated in the material (in austenite phase) after '*n*' cycles.

The variation in RS and RD upon TMC of the cold worked and annealed NiTi wire is shown in figures 8–9. It can be seen that the material is unstable upon TMC. In general, RD increases continuously with TMC. The rate of increase in RD is significant during the initial stages of cycling and it continuously decreases as the TMC progresses. The variation in RS is relatively less and is mostly confined to the initial stages of cycling. This unstable behaviour of the NiTi wire upon TMC was observed in all the samples listed in table 2.

Figure 8 shows the effect of percent cold work on the strain response of NiTi wire upon TMC. It can be seen

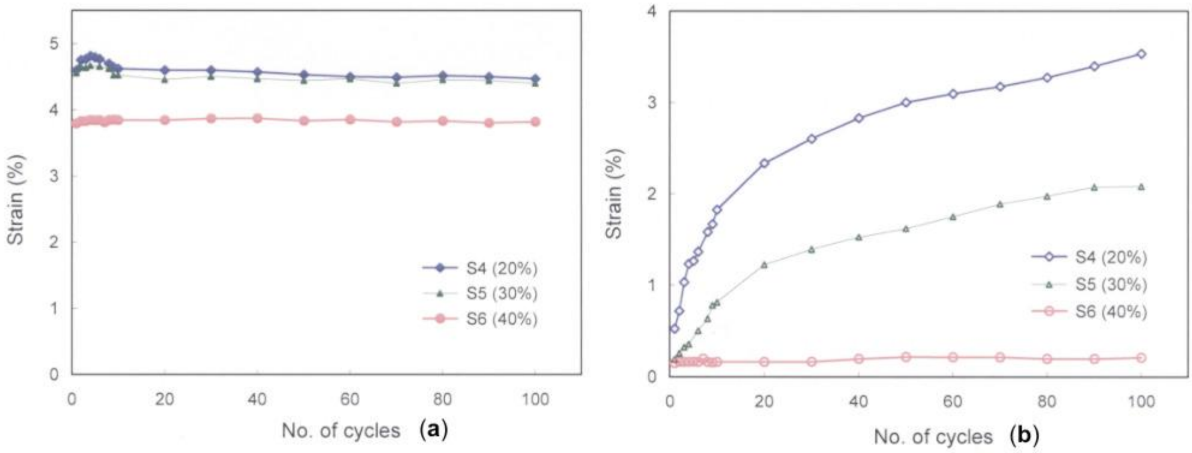

Figure 8. Variation in (**a**) RS and (**b**) RD upon TMC at 200 MPa of NiTi wires with varying cold work and annealed at 400°C.

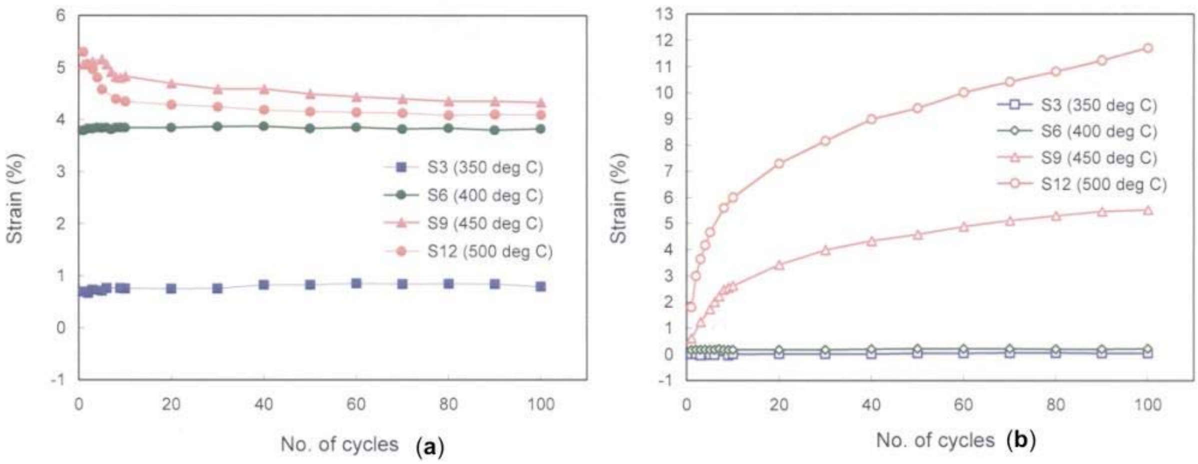

Figure 9. Variation in (**a**) RS and (**b**) RD upon TMC at 200 MPa of NiTi wires with 40% cold work and annealed at various temperatures.

that for a particular annealing temperature, the variation in RD with percent cold work is quite significant compared to that of RS. The RD was found to be higher for samples with lower retained cold work.

The effect of shape memory annealing temperature on the RS and RD of NiTi wire upon TMC is shown in figure 9. It can be seen that the decrease in shape memory annealing temperature has a similar effect on RS and RD as that of the increase in percent cold work. The increase in RD is quite significant beyond a critical range of shape memory annealing temperature. In the present set of experiments, the NiTi wires annealed above 400°C were found to be very unstable upon TMC.

The strain response of the NiTi wire during TMC has a strong bearing on the stress under which the TMC is carried out. Results show that the magnitude and rate of change in RS and RD increase with the increase in TMC stress (figures 8 and 10). Results also showed that this variation in strain response could be minimized by subjecting the wire to TMC at relatively higher stresses than that of the actual TMC stress for a few cycles. For example, the NiTi wire subjected to TMC at 300 MPa stress for 50 cycles followed by TMC at a stress of 200 MPa (figures 10–11) shows less variation with respect to RS and RD than that of wire subjected to TMC at 200 MPa alone (figures 8–9). The overall stability of the material, however, is strongly dependent on the combination of the amount of cold work and the annealing temperature. For example, though the processing parameters are different for samples S2 and S6, they exhibit similar levels of stability with respect to RD upon TMC under the present experimental conditions (table 2).

3.2b *Phase transformation characteristics*: The processing parameters were found to have significant influence on the cyclic transformation behaviour of the NiTi

Table 2. Variation in RS and RD during TMC of NiTi wires processed under varying conditions.

	TMC at 300 MPa for 50 cycles		TMC at 200 MPa for 500 cycles	
Sample	RS (%)	RD (%)	RS (%)	RD (%)
S1	4·2	4·0	3·5	0·8
S2	3·5	0·3	3·0	0·05
S3*	–	–	0·8	0·04
S3	2·5	0·2	1·4	0·03
S4*	–	–	4·5	3·5
S4	4·0	5·2	3·5	0·5
S5*	–	–	4·4	2·1
S5	4·2	2·2	3·6	0·3
S6*	–	–	3·8	0·2
S6	4·2	0·6	3·4	0·05
S7	3·8	9·7	3·4	3·0
S8	3·9	6·6	3·6	1·2
S9*	–	–	4·1	5·5
S9	4·0	5·8	3·6	0·9
S10	3·7	8·5	3·1	1·5
S11	3·8	10·5	3·4	4·3
S12*	–	–	4·3	11·7
S12	4·0	14·1	3·5	7·2

*TMC at 300 MPa not carried out and the data of TMC at 200 MPa for 100 cycles reported

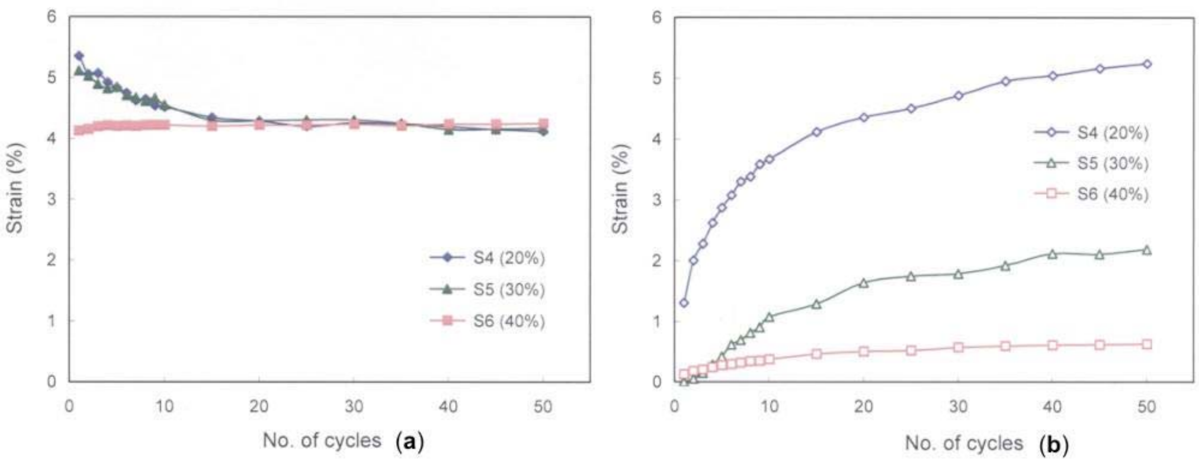

Figure 10. Variation in (**a**) RS and (**b**) RD during TMC at 300 MPa of NiTi wires with varying cold work and annealed at 400°C.

wire. In general, the increase in retained percent cold deformation and decrease in shape memory annealing temperature have a similar effect on the transformation behaviour. Though the transformation hysteresis remains almost unchanged, the TTs shift to higher temperatures with decrease in cold work and increase in annealing temperature (figure 12). Also, the slope of the curve tilts towards left with increase in retained percent cold work.

The transformation behaviour of the cold worked and annealed NiTi wire was found to be very unstable upon TMC (figure 13). Results show that the transformation width (hysteresis) decreases with the progress of TMC. The TTs in martensite phase (M_s, M_f) increase while there is no noticeable change in the austenite phase (A_s, A_f) (figure 13). The transformation behaviour tends to stabilize with increase in number of cycles of TMC, similar to that observed in RS and RD. However, the number of cycles required to reach this stage is a function of processing parameters and the stress under which the TMC is carried out. It can be seen that the stabilization of the transformation behaviour is accelerated if the TMC is carried out at higher stresses followed by TMC at lower stresses (figure 14).

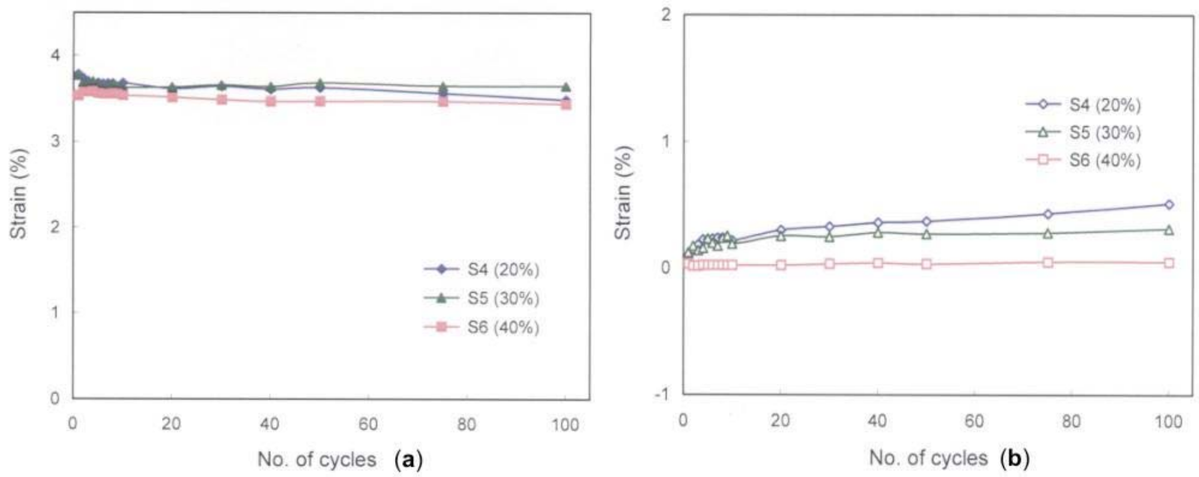

Figure 11. Variation in (**a**) RS and (**b**) RD during TMC at 200 MPa following TMC at 300 MPa for 50 cycles of NiTi wires with varying cold work and annealed at 400°C.

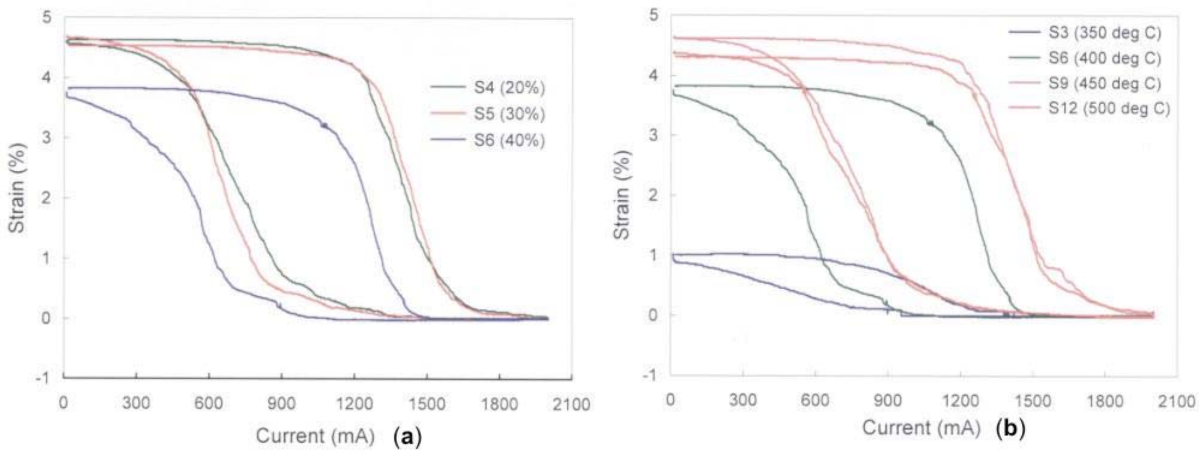

Figure 12. Transformation hysteresis of NiTi wires during 100th cycle of TMC at 200 MPa: (**a**) varying cold work and annealed at 400°C, and (**b**) 40% cold work and varying annealing temperature.

4. Discussion

The basis for the shape memory effect in SMAs is the presence of twinned structure in martensite phase. On application of load, detwinning takes place resulting in macro deformation in the material which is recoverable completely/partially on heating to austenite phase. It is well known (Humbeeck *et al* 1990; Saburi 1998; Miller and Lagoudas 2001; Bhaumik 2008) that optimization of the processing parameters viz. the retained cold work and shape memory annealing temperature/time is essential to obtain superior shape memory effect. Cold work introduces high density of random dislocations in the material resulting in strengthening of the martensite phase. This facilitates the deformation in martensite phase preferably by detwinning rather than slip mechanism. However, these random dislocations impede the mobility of the twin boundaries and thus restrict shape memory effect. This is evident by the fact that the cold worked material has similar stress–strain behaviour to that of the conventional engineering materials (figure 3) and shows very poor or no shape memory effect. The worked material requires to be annealed at moderate temperatures (below recrystallization temperature) to achieve shape memory effect. During annealing, rearrangement of random dislocations takes place resulting in formation of cells of relatively dislocation free areas surrounded by dislocation networks. The martensite twins are glissile within the dislocation free areas, thus facilitating shape memory effect. The dislocation networks provide the necessary strength to the martensite phase to avoid deformation of the material by slip mechanism.

It has been shown in the present study that irrespective of the processing parameters chosen, NiTi wire shows unstable behaviour upon TMC (figures 8–10). The RS and RD change continuously with the progress in TMC.

The magnitude of change, however, is dependent on the processing parameters as well as the stress under which the TMC is carried out. Studies (Perkins and Sponholz 1984; Humbeeck *et al* 1990; Humbeeck 1991; Stalmans *et al* 1991; Jiang *et al* 1997; Liu *et al* 1999) have shown that the RD results in the material because of two major factors: (a) irreversible plastic deformation in the austenite phase, and (b) stabilization of the martensite/austenite phase. The irreversible plastic deformation in the austenite phase is believed to be due to generation of defects/dislocations in the microstructure during TMC (Perkins and Sponholz 1984; Stalmans *et al* 1991; Jiang *et al* 1997; Liu *et al* 1999; Rong *et al* 2001; Otsuka and Ren 2005). These defects/dislocations in turn are responsible for stabilization of certain volume fraction of martensite/austenite phase in the material that does not take part in transformation during TMC. The density of defects/dislocations increases with the number of cycles and so does the volume fraction of stabilized martensite/austenite. The RD increases with increase in the irreversible deformation in the austenite phase and stabilization of martensite phase. The increase in volume fraction of stabilized martensite phase also results in decrease in the RS since less amount of martensite is now available for providing transformation strain.

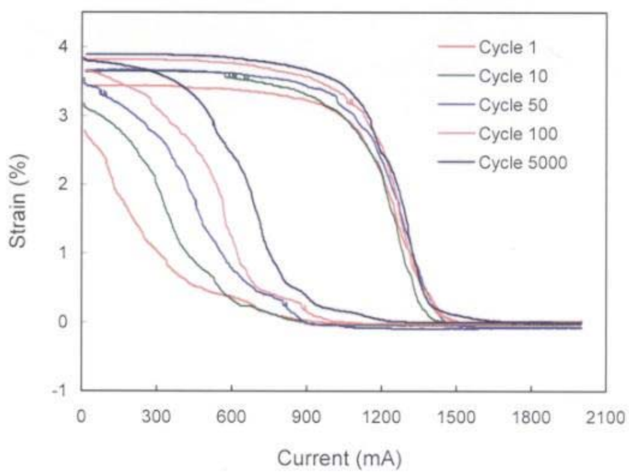

Figure 13. Transformation hysteresis during TMC at 200 MPa of NiTi wire with 40% cold work and annealed at 400°C.

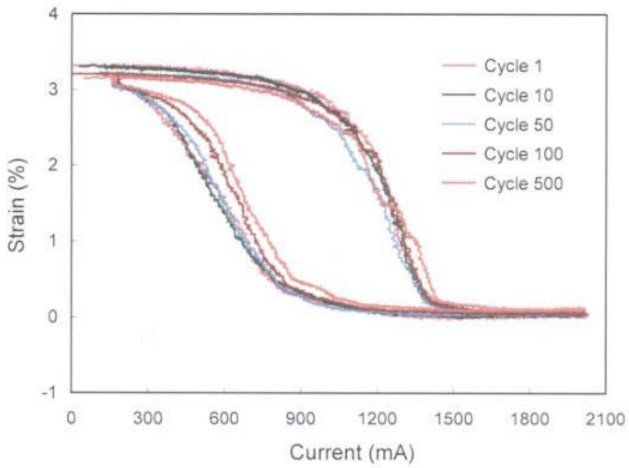

Figure 14. Transformation hysteresis upon TMC at 200 MPa of NiTi wire with 40% cold work and annealed at 400°C; initially subjected to TMC at 300 MPa for 50 cycles.

Since the unstable behaviour of NiTi wire during TMC is associated with the microstructural changes, it is expected that TMC would influence the stress–strain behaviour as well. It can be seen that the yield strength in the martensite phase increases with TMC and the nature of stress–strain curve approaches towards that of the cold worked material with the progress of cycling (figure 6). This is an indirect evidence of defect generation in the material during TMC.

Results show that the magnitude and rate of change in RS and RD during TMC is highly dependent on the properties of the starting material. For example, the RD in the material can be reduced substantially by increasing the percent cold work or by lowering the shape memory annealing temperature (figures 9–10). This means that retention of defects introduced during cold working is helpful in reducing the defect generation in the material during TMC. In other words, a material with high yield strength shows a more stable behaviour upon TMC than that of a material with low yield strength. Results also show that the degree of cold work and the annealing temperature can be selected depending on the shape memory strain to be utilized and the degree of stability required during TMC (table 2). However, irrespective of any combination, the material behaves in a very unstable manner with respect to RD, when annealing is carried out above 400°C.

The characteristic transformation temperatures (TTs) of the SMA largely depend on the balance between chemical driving energy of transformation (ΔG^{c}) and the opposing non-chemical energy (ΔG^{nc}). The ΔG^{nc} is essentially the elastic and irreversible energies that are built up during martensitic transformation and generation of defects in the structure (Perkins 1975; Jiang *et al* 1997). The increase in the TTs observed (figures 12–13) during TMC in the martensite phase is due to increase in ΔG^{nc} as a result of introduction of dislocations in the material (Stachowiak and McCormick 1988). The dislocation structure developed during TMC facilitates nucleation of preferred variants in the direction of applied stress, thus raising M_s and M_f. This defect generation during TMC is consistent with the observations made in the case of RS and RD. However, when the material is stable, no or negligible variation is observed in the transformation behaviour upon TMC (figure 14).

It has been shown that the stability of the material can be improved to some extent through appropriate combination of cold work in the material and the annealing temperature (figures 8–9). It may, however, be noted that irrespective of any processing conditions, the material is unstable for use where repetitive TMC is a requirement. Though RS stabilizes within the initial few cycles, the RD continuously increases with the progress in TMC. The rate of increase is high to start with and it decreases continuously with the increase in the number of TMC cycles. The material tends to behave in a relatively stable manner after a large number of TMC cycles. Keeping in view the close proximity between defect generation in the material and the change in RS/RD, this implies that at a particular stress level, with continued TMC, the material attains a certain defect density such that further generation of defects in the material is diminished, and the material starts behaving in a relatively stable manner with minimal changes in RS and RD. Following the same argument, it can also be stated that the defect generation in the material can be accelerated by increasing the stress level during TMC. For example, the stress–strain plot of NiTi wire specimen, S6, shows a similar behaviour upon TMC at 300 MPa for 50 cycles to that of TMC at 200 MPa for 1000 cycles, indicating a comparable defect structure in the material in both the cases (figure 7). This implies that the wire that shows relatively stable behaviour after 1000 cycles of TMC at 200 MPa would show a similar level of stability from the first cycle onwards if it is given a prior stabilization treatment through TMC at 300 MPa for as low as 50 cycles (table 2).

The results of the present study show that to obtain superior shape memory effect in NiTi wire, appropriate selection of percent cold work and shape memory annealing temperature is important. The selection of these parameters is guided by the application requirements. It has also been shown that though the processing history has significant influence on the mechanical and functional behaviour of NiTi wire as a whole, an additional post-processing stabilization treatment would be necessary for applications where stable strain response upon repetitive TMC is of prime concern.

5. Conclusions

The processing history of NiTi wire has significant influence not only on the mechanical properties but also on the functional properties. It assumes more significance when the stability of the material upon TMC is of great concern. In this context, the following conclusions can be drawn from the present study.

(I) The mechanical properties of NiTi wire can be altered significantly by varying retained cold work and the shape memory annealing temperature, individually or in combination. But to achieve superior shape memory effect in the material, appropriate selection of both the parameters is important.

(II) Irrespective of any processing conditions, NiTi wire is unstable when subjected to TMC. The RS and RD continuously change with the progress in the TMC, and the behaviour of the wire tends to stabilize only after a large number of cycles. The magnitude of change in RS/RD is dependent on the stress at which the TMC is carried out and also, the mechanical properties of the wire.

(III) The processing parameters chosen to obtain superior shape memory effect in NiTi wire need not necessarily result in a material which would show stable behaviour upon TMC. An optimum selection is necessary to minimize the instability in terms of minimal change in RS and RD upon TMC. The study shows that selection of processing parameters must be based on the transformation strain and the stability that are required during the TMC.

(IV) NiTi wires with high retained cold work show relatively better stability upon TMC. Such wires may eventually have less available transformation strain.

(V) For a stable strain response during TMC, the NiTi wire requires a post-processing stabilization treatment. Study shows that a few cycles of TMC at higher stresses than that of the application stress could be one such stabilization process.

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