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THE EFFECTS OF PSEUDOELASTIC PRESTRAINING ON THE TENSILE BEHAVIOUR AND TWO-WAY SHAPE MEMORY EFFECT IN AGED NiTi

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Abstract—Underaged Ti–50.85at.% Ni wire was pseudoelastically prestrained between 2 and 8%. A second deformation to the same strain was found to occur at stresses some 60 MPa lower than the flow stresses measured during the prestraining. Deformation beyond the original prestrain required increasing the stress to the original levels. This created two distinct plateaus in the stress–strain behaviour. Related shifts in the transformation temperatures were observed during cooling and heating of the wire under constant load. Ageing the specimens at 160°C (433 K) after the first cycle erases the effect completely. The results give no evidence that either changes in the dislocation structure or sheared precipitates are responsible for this phenomenon. It is suggested that small martensite nuclei persist above A_f after unloading which favour the formation of convenient martensite orientations during subsequent deformation. These martensite orientations also form during stress-free cooling of the wires causing a strong two way shape memory effect.

Résumé—Un fil de titane–nickel à 50,85% en atomes de nickel, sous-vieilli, a été prédéformé pseudo-élastiquement entre 2 et 8%. Une seconde déformation jusqu'au même taux a lieu pour des contraintes inférieures d'environ 60 MPa aux contraintes d'écoulement mesurées pendant la prédéformation. Si l'on veut déformer au delà de la prédéformation initiale, il est nécessaire d'augmenter la contrainte jusqu'aux valeurs initiales. Ceci crée deux paliers différents dans la courbe contrainte–déformation. Les écarts correspondants dans les températures de transformation ont été observés pendant le refroidissement et le chauffage du fil sous charge constante. Le vieillissement des échantillons à 160°C (433 K) après le premier cycle fait disparaître complètement cet effet. D'après les résultats, ni les modifications de structure des dislocations, ni celles des précipités cisaillés ne seraient responsables de ce phénomène. On suggère que de petits noyaux de martensite persistent au dessus de A_f après la décharge, ce qui favorise la formation d'orientations convenables de la martensite pendant la déformation ultérieure. Ces orientations de la martensite se forment aussi pendant le refroidissement des fils sans contrainte, ce qui cause un effet important de mémoire de forme cans les deux sens.

Zusammenfassung—Eine unteralterte Ti–50,85at.-% Ni Legierung wurde im pseudoelastischen Bereich Dehnungen zwischen 2 und 8% ausgesetzt. Bei einer zweiten Verformung wurde beobachtet, daß die notwendige Spannung zum Erreichen der Dehnung des ersten Versuchs etwa 60 MPa niedriger lag. Bei einer Verformung über die Dehnung des ersten Versuchs hinaus steigt die dafür notwendige Spannung wider auf das ursprüngliche Niveau an: Es entstehen zwei unterschiedliche pseudoelastische Plateaus in der Spannungs-Dehnungskurve. Bei einer martensitischen Umwandlung unter konstanter äußerer Last zeigten sich parallel dazu Verschiebungen der Umwandlungstemperaturen nach der (reversiblen) Vorverformung der Probe im pseudoelastischen Bereich. Wenn die Proben nach der ersten Verformung für 1 Sekunde auf 160°C (433 K) erwärmt werden, verschwindet der Effekt vollständig. Es kommen daher weder gescherte Ausscheidungsteilchen noch Änderungen in der Versetzungsstruktur als Erklärung des Phänomens in Frage. Es wird vorgeschlagen, daß kleine Martensitkeime auch noch oberhalb der A_f Temperatur erhalten bleiben und bei der zweiten Verformung die Bildung günstig zur Spannungsrichtung liegender Martensitorientierungen fördern. Dieselben Orientierungen bilden sich auch bei der Abkühlung der Probe ohne äußere Spannung und bewirken dabei einen großen Zweiwegformgedächtniseffekt.

1. INTRODUCTION

The subject of this paper is an unusual phenomenon found in pseudoelastically prestrained NiTi wire that has been solution treated and underaged prior to prestraining. Figure 1 demonstrates the effect: here a wire is pseudoelastically deformed to 4% strain, unloaded, and is then deformed to 8%. During the second loading, two distinct yield points and stress plateaus are observed: initial yielding occurs at a lower stress than during the first cycle, but when the deformation strain reaches that of the first cycle, the stress abruptly increases to the level of the original plateau. The purpose of these investigations is to understand this unusual yield drop effect and to explain its origins.

Yielding of pseudoelastic NiTi causes a stress induced martensitic transformation of the high temperature β-phase (B2) to a monoclinic B19′

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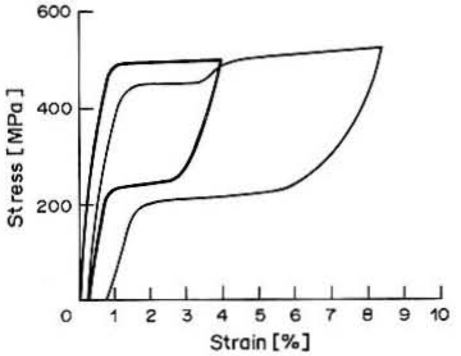


Fig. 1. The yield drop phenomenon: an underaged NiTi wire is deformed to 4% and unloaded in the pseudoelastic range (shown by the heavy line). A second pseudoelastic deformation to 8% shows a discontinuity in flow stress at 4%.

martensite. In polycrystalline material up to 8% strain can be accommodated by this transformation. Since the martensite becomes unstable when the stress is removed, the strain is almost completely recovered during unloading. Tensile tests of pseudoelastic conditions show three deformation stages [1]: the elastic deformation ϵ_{el}, an initial, reversible yielding and deformation plateau due to the stress inducement of martensite ϵ_{ps} (pseudoelastic deformation), then a third irreversible yield due to dislocation movement ϵ_{pl} (true plastic deformation). The total strain ϵ_t is then

$$\epsilon_t = \epsilon_{el} + \epsilon_{ps} + \epsilon_{pl}. \tag{1}$$

The stress level of the pseudoelastic plateau is primarily determined by the difference between the test temperature and the martensite start temperature (M_s) with the plateau stress increasing as the temperature difference increases according to the Clausius–Clapeyron equation. In addition, the crystal orientation of the β-phase affects the stress level and extent of transformation induced strain [2].

Ni-rich NiTi alloys are well known to be unstable and precipitation sequences during ageing are described in several papers [3–6]. Specimens aged after solution treatment, or annealed immediately after cold working, exhibit a change in transformation behaviour during cooling from the B2 (austenite) to B19′ (martensite) sequence described above to the sequence: B2 to incommensurate B2 to the rhombohedral R-phase and then finally to B19′ [7, 8]. Tensile testing such conditions shows a four stage deformation process: first an elastic deformation then a reversible strain ϵ_R of up to 1% due to the reorientation of the R-phase, then the strain plateau related to stress inducing the martensite, and finally permanent deformation due to slip processes [9–11]

$$\epsilon_t = \epsilon_{el} + \epsilon_R + \epsilon_{ps} + \epsilon_{pl}. \tag{2}$$

In the temperature range below the austenite finish temperature (A_f) an increasing shape memory strain ϵ_{sm} occurs, which is reversible during heating above A_f. Below the austenite start temperature (A_s) ϵ_{sm} replaces ϵ_{ps} and the strain is described by

$$\epsilon_t = \epsilon_{el} + \epsilon_R + \epsilon_{sm} + \epsilon_{pl}. \tag{3}$$

Above M_d (the temperature above which martensite cannot be stress induced) the total strain is simply

$$\epsilon_t = \epsilon_{el} + \epsilon_{pl}. \tag{4}$$

2. EXPERIMENTAL

A Ni–Ti alloy with 50.85at.% Ni was melted in vacuum, hot swaged and cold drawn 40% to a 0.47 mm diameter wire. The wires were then solution treated for 30 min at 850°C (1123 K) in a Helium atmosphere, water quenched and aged in a salt bath at 325°C (598 K) for various times. Transformation temperatures were measured in the unstressed state using Differential Scanning Calorimetry (DSC). Transformation temperatures and strains were also measured by applying a 225 MPa stress to the wires and then thermally cycling in an ethanol bath while monitoring strain. Tensile testing was done using an extensometer in a temperature chamber.

3. OBSERVATIONS

The presence and magnitude of the stress drop are not dependent upon the total strain during prestraining. Tensile tests were conducted on wire prestrained to 2, 4, 6 and 8% [see Fig. 2(a–d)]. The plateau level during the second deformation always occurred at the same lower stress level.

The largest stress drop between the first and second tensile test was found in NiTi that was solution

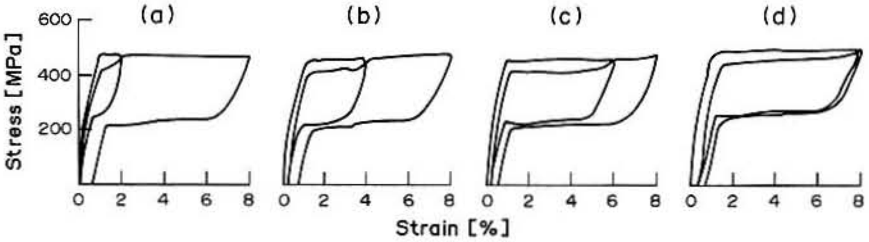


Fig. 2. The effect of changing the prestrain level on the yield drop phenomenon. (All tests were at room temperature, a new specimen was used for each test.)

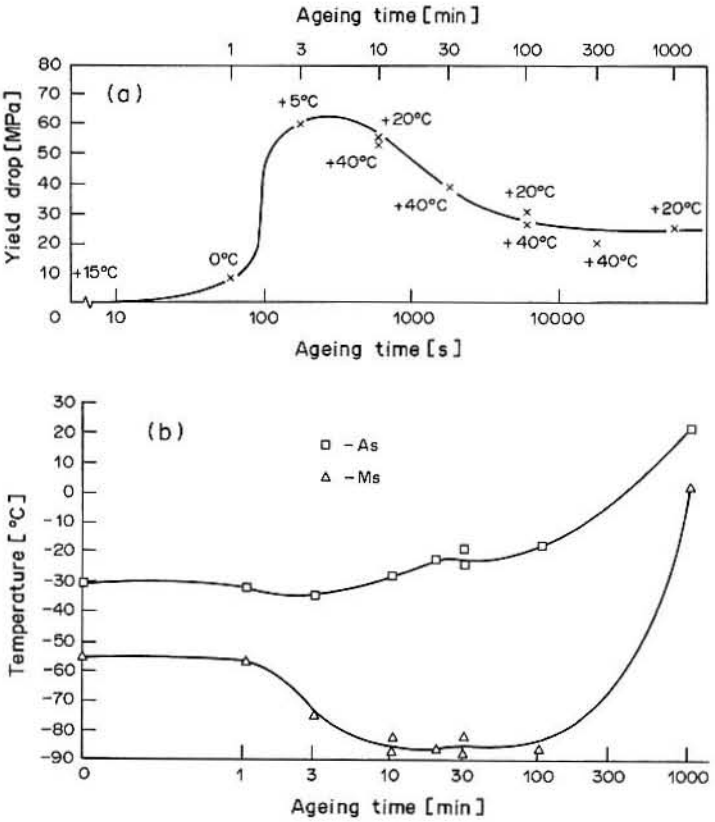


Fig. 3. (a) The effect of ageing time at 325°C on the magnitude of the yield drop (measured at 2% strain). The test temperature is indicated for each specimen. (b) M_s and A_s vs ageing time as measured by DSC.

treated after cold working and aged at 325°C (598 K) for only 3 min [Fig. 3(a)]: further ageing caused a decrease in the stress drop from more than 60 MPa to less than 30 MPa after 1000 min.† The same alloy was tested after cold drawing 42% and then directly annealing at 350, 400 and 450°C (623, 673 and 723 K). Even though the austenitic strength levels were very high, the yield drops were only 15, 30 and 15 Mpa respectively for the three temperatures. All further experiments reported here were done with the NiTi wire aged for 10 min at 325°C (598 K) after a solution treatment at 850°C (1123 K).

During repeated pseudoelastic deformations to 4% a continuous decrease in the plateau stress is observed (Fig. 4) with the largest decrease occurring between the first and second cycles. The plateau level during unloading decreases as well, but the effect is comparatively small. If, after repeated deformation to 4%, the specimen is elongated to 8%, the stress will increase to the original plateau upon reaching 4%; unlike cyclic hardening and softening in conventional metals, only a portion of the stress–strain curve is affected.

†Since the M_s in NiTi is known to shift during ageing [Fig. 3(b)], it was necessary to change the testing temperatures during these tests in order to keep the deformation in the pseudoelastic range. It will be shown in Fig. 7 that such variation does not significantly influence the yield drop.

A tensile curve of a wire pseudoelastically prestrained two times (to 4% and then to 8%) is shown in Fig. 5: the third stress–strain curve now exhibits three plateaus: the first (A) is caused by the two deformations to 4% (the shift to 4.8% is due to a partial shape memory strain ϵ_{sm} which remained after the second deformation); the second (B) corresponds to the second prestrain to 8%; the third (C) is on the original loading plateau. This result is important in that it shows that the stress drop is not simply due to the amount of the prestraining, but that there is a memory involved: *the material "knows" how far, and how many times, it has been prestrained.* Given only the third cycle curve of Fig. 5, one could deduce that the material was prestrained to 4 and to 8% prior to testing. (It should be remarked that the slight inflection in the elastic range above 250 MPa is due to the R-phase reorientation causing a strain ϵ_R up to 1%).

It is also interesting to look at partial unloading. If a specimen is strained to 6% for example, and then partially unloaded to 4%, reloading shows a plateau at the same level as it would have after complete unloading (Fig. 6). (The second unloading plateau

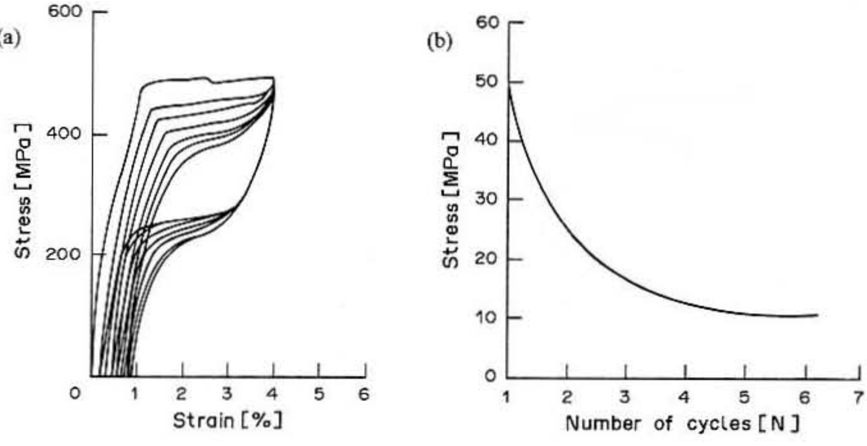


Fig. 4. (a) Pseudoelastic cycling to 4% causes a continuous reduction in the flow stress; subsequent straining beyond 4% returns the original flow stress. (b) Yield drop vs number of cycles.

level decreases slightly when the 4% strain is achieved but not always as clearly as in Fig. 6.) A third loading shows the plateau at the level of the second deformation after partial unloading.

The effect of test temperature on the yield drop is rather small (Fig. 7). It can be observed at all temperatures in the pseudoelastic range of the alloy. If the temperature is too low (below A_f), the strain is not fully recoverable without heating and the effect cannot be determined [see equation (3)]. Above M_d, there is obviously no effect.

If the specimen is cooled below M_s after prestraining, randomly oriented martensite variants can be expected to form, which might be expected to delete the effect; but this, too, was found to have no effect on the phenomenon.

Ageing at room temperature after prestraining (up to 42 h) has no effect on the second cycle behaviour, this indicates that strain ageing types of mechanisms do not play a role. On the other hand, modest heating between the first and second deformations did affect the magnitude of the drop. Figure 8 shows the magnitude of the yield drop after prestraining 4% and heating to various temperatures for 5 min. Even at 60°C (333 K) the yield drop becomes less pronounced; heating to 150°C (423 K) completely erases the effect. Heating times as short as 1 sec above 150°C were sufficient to "reset" the material implying that the deletion process is athermal (Fig. 9).

Differential Scanning Calorimetrie (DSC) measurement give evidence of an R-phase transformation at 1°C (274 K) and M_s at -89°C (184 K). Comparison of DSC measurements between a new specimen and specimens prestrained to 2, 4, 6 and 8% show the development of a shoulder on the warm side of the martensite peak, with the shoulder becoming more pronounced with increasing prestrain [see Fig. 10(a)]. After 8% prestrain the shoulder peak becomes dominant and it actually appears to be the primary peak. The effect of previous deformation on the shape of the peaks of the reverse martensite to

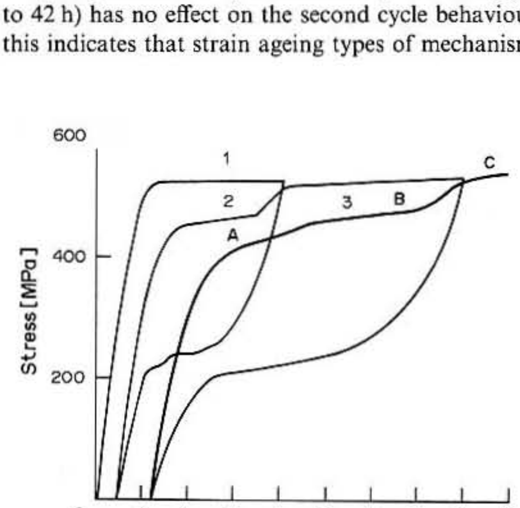


Fig. 5. The underaged NiTi wire is prestrained twice: curve 1 shows the first prestraining to 4% and curve 2 shows the second prestraining to 8%. A third deformation (heavy line) then exhibits two yield drops (A and B) before the original flow stress is achieved (C).

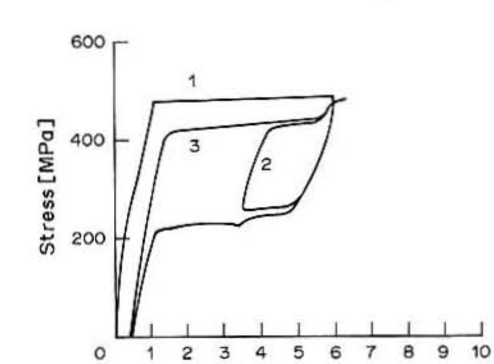


Fig. 6. The prestraining (curve 1) is interrupted during unloading and the specimen is again deformed (curve 2) to show a yield drop. A third deformation occurs at the same reduced flow stress (curve 3).

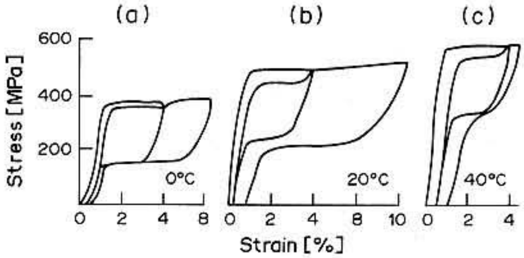


Fig. 7. The effect of test temperature on the yield drop phenomenon.

austenite transformation during heating was negligible [Fig. 10(b)], though peak intensity was decreased. The transformation peaks of a specimen prestrained to 8% and aged at 160°C (433 K) for 60 min before DSC testing is given in Fig. 10(c): the peak intensity does not change significantly after the ageing treatment. It should be noted that similar "splitting" of the martensite peak has been observed after partial thermal cycling [12, 13], but has not been fully explained.

Measuring transformation temperatures by monitoring the wire elongation as function of temperature under constant load shows a "step" on cooling of prestrained wires. Two examples are shown in Fig. 11: a specimen prestrained 2% starts deformation during cooling under the constant stress of 225 MPa at 22°C (295 K) due to the R-phase transformation (ϵ_R). At -35°C (238 K) a first martensitic transformation causes an additional strain of about 1% and at -44°C (229 K) another 4% strain completes the transformation. The total strain is fully recoverable during heating between 8 and 11°C (281 and 284 K), and the strain associated with the R-phase is recovered at 30°C (303 K). The dotted line shows the behaviour of the same specimen after heating to 160°C (433 K): the two step elongation during the martensitic transformation has changed to one step at the lower temperature. After 8% prestrain [Fig. 11(b)] the curve looks similar but the first elongation step is the larger one with almost 4%, the second step smaller with an additional 1% strain.

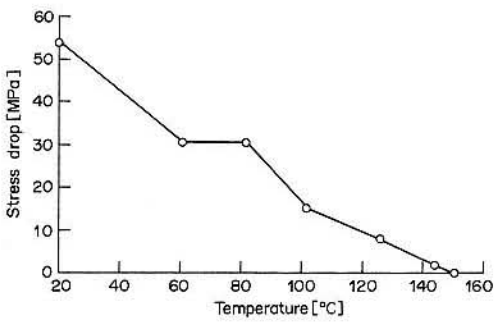


Fig. 8. The effect of 5 min ageing at various temperatures on the magnitude of the yield drop.

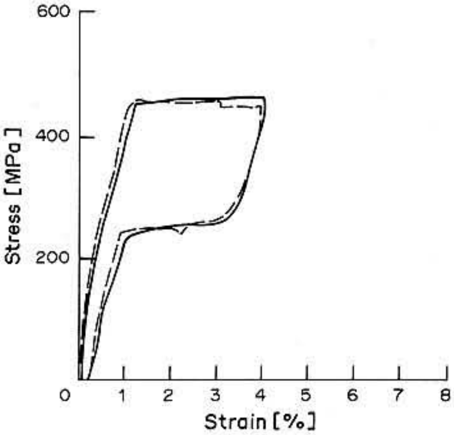


Fig. 9. The stress–strain–curve of a wire prestrained to 4% (solid line), then heated to 160°C for 1 s, then again strained to 4% at room temperature (dotted line). The stress drop is eliminated by the heating.

Again, heating to 160°C (433 K) erases the effect (dotted line). The important point is that a portion of the transformational strain after prestraining occurs at higher temperatures than in virgin wire, and that the amount of this "premature" transformation is related to the amount of prestrain. The direction of this shift is expected: an increase in M_s is equivalent to a decrease in the stress needed to induce martensite, and thus the flow stress drop in a stress–strain curve manifests itself as a temporary increase in M_s. The yield drop can be quantitatively related to the M_s shift through the Clausius–Clapeyron equation:

$$\frac{d\sigma}{dT} = \frac{\Delta H \cdot \rho}{\epsilon \cdot T_0} = 9.5\ \mathrm{MPa/K} \qquad (5)$$

(with $\Delta H = 29.3$ Nm/g, $\rho = 6.5$ g/cm^3, $\epsilon = 0.08$ and $T_0 \approx 250$ K). Thus at 55 MPa we expect a 6 K shift. The measured shift was about 7 K (Fig. 11).

Final experiments were made by stretching wires to 4, 8 and 9% at room temperature. After unloading, a two-way shape memory effect was observed during cooling to -160°C (113 K). The magnitude of the two-way effect after a 4% deformation was 1.5% during cooling and heating without external stress. A prestrain of 8% creates a two-way of 2.1%, 9% prestrain increases the two way effect to 2.6%. The effect is quite stable even after repeated cooling and heating up to 90°C (363 K) (measured up to 7 times). Heating the specimen to 160°C (433 K) erases the yield drop but curiously has no influence on two-way shape memory [Fig. 12(a) and (b)].

4. DISCUSSION

The observations discussed above are unusual and difficult to fully explain. Before discussing various possibilities it is useful to quickly review some of the key characteristics:

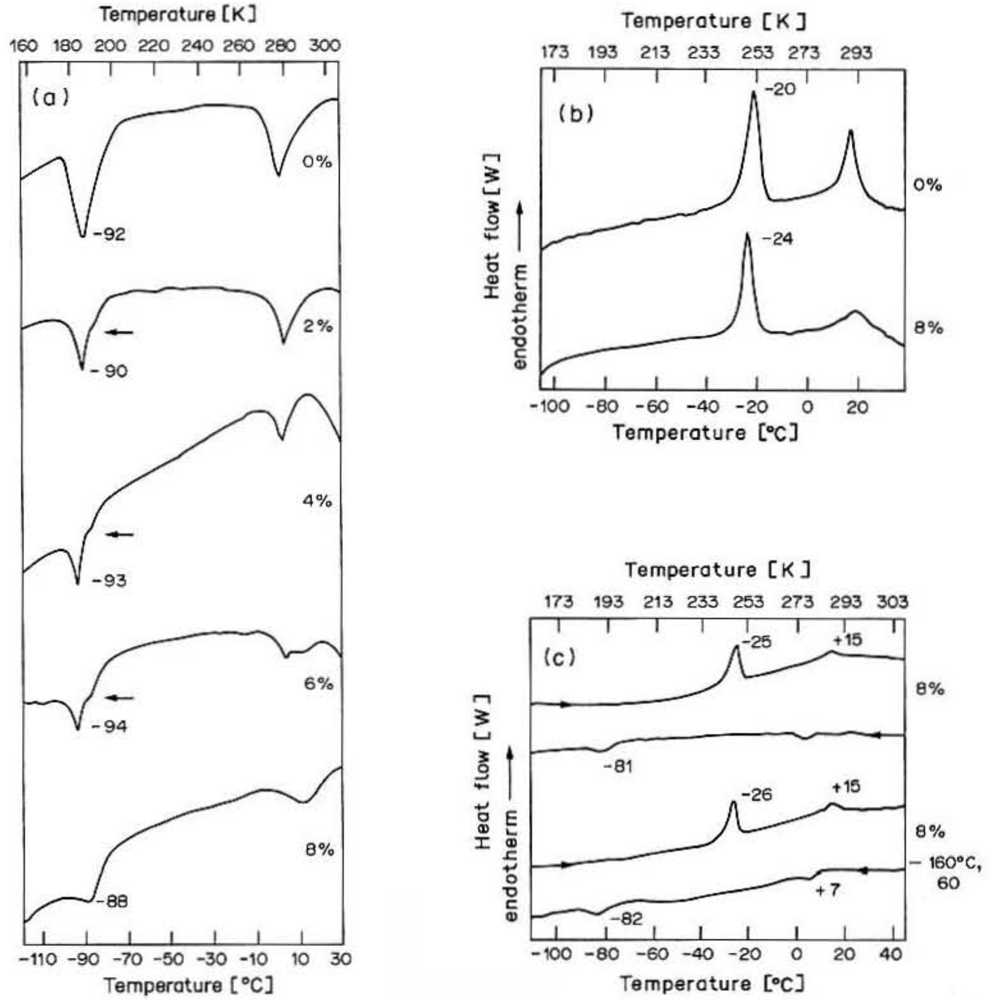


Fig. 10. (a) DSC measurements during cooling of the Ni–Ti wire after different previous deformation treatments. Temperatures of peak maxima indicated. Development of a shoulder on the warm side with increasing prestrain is indicated by arrows. (b) DSC curves during heating of an unstrained specimen and one prestrained to 8%. (c) DSC curves during cooling of a specimen prestrained to 8% and of the same specimen heated to 160°C for 1 h after prestraining to 8%.

1. The change in the plateau stress can be made to occur anywhere along the stress induced plateau depending on the amount of prestraining; it is, therefore, not associated with a martensite-to-martensite transformation such as it is found in Cu–Al–Ni, nor is it associated with R-phase [9–11, 14, 15].

2. Repeated prestraining results in a further decrease of the flow stress but does not extend the first plateau to higher strains (Fig. 2).

3. Modest heating after prestraining completely erases the yield drop phenomen (Fig. 8) but does not effect the two way shape memory effect (Fig. 12) and the DSC curves [Fig. 10(c)].

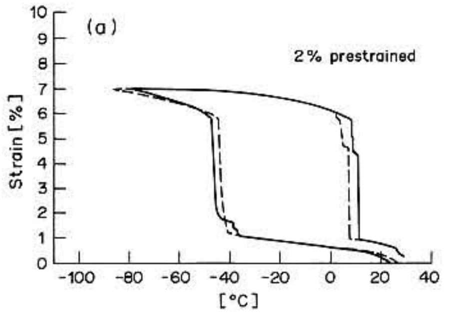
4. The resetting process is apparently athermal in nature.

5. The stress drop is greatest in underaged conditions (Fig. 3) but was not observed after solution treatment only.

6. Measurements of M_s in prestrained material indicate: a portion of the material has an M_s that is shifted to higher temperatures [Fig. 10(a) and 11(b)] than in the virgin material.

7. The material has a memory of sorts: by examining a tensile curve it is possible to determine how many times it has been prestrained, and how far (Fig. 5).

An increased dislocation density cannot account for the yield drop phenomenon: all deformation was in the pseudoelastic temperature range where the strain is caused by stress induced martensitic transformation. The small irreversible strains that remain after unloading disappear upon heating—we cannot exclude, however, the possibility that some small amount of true plastic deformation ($<0.2\%$) occurs. Dislocations have been observed [15–17] to result



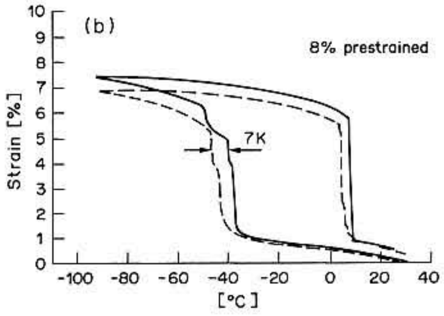


Fig. 11. (a) Heating and cooling under constant stress of 225 MPa of a prestrained wire (2%) (full line) and of the same wire after heating to 150°C for 5 min (dotted line). (b) Same test with a wire prestrained 8%.

from thermal cycling, but these tend to decrease M_s with the consequence that a second yielding should show the plateau at a higher value than during the first deformation. These same accommodation dislocations can be expected to form during pseudoelastic cycling. It could be that a dislocation network favourable for growth of selected martensite variants is left behind causing the yield phenomenon and the two way shape memory effect [18, 19]. These could in principle result in a lower plateau stress. The dislocations might even be expected to disappear by the modest heatings (Fig. 8) due to a change in the stability of partial dislocations through a change in the modulus [20]. But any dislocation rearrangement is expected to have a time dependence which is in contradiction to the observation that the deletion process is athermal. Thus this explanation is considered unlikely for the yield drop phenomenon.

Since the yield drop is observed to be largest in underaged conditions it may be that precipitates are sheared by the martensite, making subsequent growth along the same route easier. Such shearing could give the type of "mechanical memory" shown in Fig. 5: the second advance of martensite is easier only until the plates again meet unsheared particles. But to reset the wire would require that the precipitates are repaired and again we would expect this to be a time dependent process.

It is also possible to imagine that a deformation process could decrease local ordering. It has been speculated that M_s was suppressed during neutron irradiation of NiTi [21] due to such a disordering. But both this experiment and theoretical work indicate that such a disordering would decrease M_s and therefore increase the plateau stress. Furthermore, Fig. 4 shows a decreasing hysteresis; it is generally thought that disordering increases hysteresis [22]. So, this explanation can also be considered unlikely.

Miyazaki and Wayman [23] have found that R-phase plates are aligned by prestraining, and that the plate will continue to form in a biased way even after heating above T_R (the temperature of R-phase transformation). But if the material is heated above the incommensurate/commensurate (I/C) temperature and then cooled below T_R, the variants will form in a randomly oriented way—the material is reset by forming commensurate B2. The implication is that the incommensurate structure has a memory of a sort, and guides the selection of R-phase variants. A similar mechanism could occur in this case, but

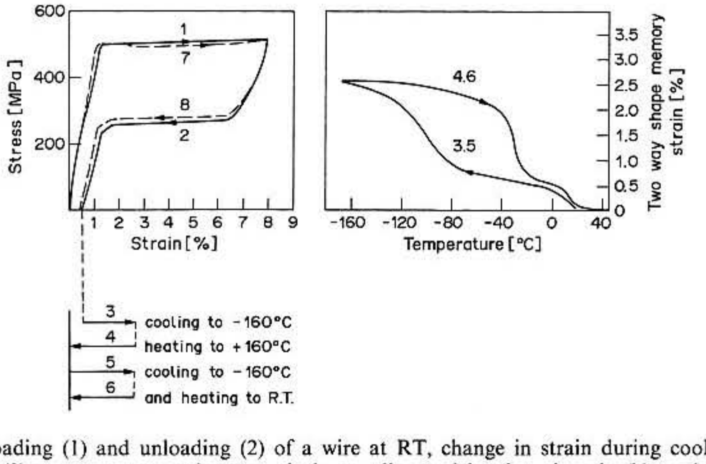


Fig. 12. Loading (1) and unloading (2) of a wire at RT, change in strain during cooling to −160°C (unloaded) (3), temperature–strain–curve during cooling and heating given in (b) strain change during heating to +160°C (4) and cooling to −160°C again (5); heating to RT again 16); dotted line: loading (7) and unloading (8) again.

exactly what the mechanism of memory is, is not known. One would expect, however, the resetting of the stress–strain curve to be better defined at the I/C temperature, instead of spread out between room temperature and 160°C (433 K) as shown in Fig. 8.

It has often been claimed that small martensite nuclei can persist above A_f, and that these are responsible for inducing the two-way effect [24]. It is possible to have the same effect here, where some residual martensite embryos are not reverted during unloading, and remain to act as nuclei for the second advance. It could than be supposed that the preferred orientation of these nuclei makes martensite transformation along these same directions easier. This necessitates, however, that the entire pseudoelastic plateau is nucleation controlled—otherwise a second prestraining would extend the lower stress region, not simply reduce its stress level as is shown in Figs 4 and 5. We also must speculate that these martensite nuclei are somehow tied up, or retained by the coherency strain fields surrounding the underaged precipitates, as proposed by Nishida [3, 4]. While the yield phenomenon can be explained by the theory of persisting martensite nuclei it cannot explain why the large two-way effect does not disappear after heating to 160°C (433 K) and why the DSC curve does not change after ageing at 160°C. Thus, one would have to assume that there are two different mechanisms working together:

1. Persisting martensite nuclei are responsible for the yield phenomenon. They seem to be favoured by the precipitations in the underaged NiTi causing the yield phenomenon to be largest after a short ageing treatment.
2. A small number of dislocations caused by irreversible plastic deformation even at strains around 8% favour the formation of special martensite orientations during cooling and are responsible for the two-way shape memory effect during cooling and heating of the prestrained wire.

5. CONCLUSIONS

The yield phenomena can be best explained by the theory of persisting martensite nuclei which remain in the austenite matrix after unloading the wire even above the A_f temperature measured in the virgin material. This is favoured by precipitations in the underaged NiTi. In good correlation between our results and previous literature the two-way effect is caused by residual dislocations due to a small amount of irreversible plastic deformation even in the plateau region of the stress strain curve and will not disappear athermal. The interaction of martensite plates with precipitates of a distinct diameter and with a small amount of dislocations can explain the whole phenomenon.

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