



## RELATION BETWEEN THE THERMOMECHANICAL HISTORY AND THE MECHANICAL RESPONSE OF A NiTi MEMORY SHAPE WIRE

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**Abstract.** *A NiTi wire was subjected to a sequence of thermomechanical loading and heating experiments as part of an experimental program for the study of the characteristics of thin memory shape elements, carried out at the Military Institute of Engineering. The experiments included stretching the wire in the austenite range at temperatures between 25° C and 60° C. Shape recovery was partial after unloading, except for loading at 60° C. The wire was heated through two temperature ranges after unloading at 25° C to analyze the material reaction under a partially recovered condition. The results show a partial form recovery during heating as indicated by the development of a recovery tensile load. The load increases continuously with temperature up to a limit given by the initial residual deformation and the maximum heating temperature. Moreover, the results show that austenite to martensite phase transformation, and the associated capacity of pseudoelastic deformation and form recovery, does not take place on consecutive loading at 25° C. Phase transformation and shape recovery are restored after loading and complete form recovery at 60° C.*

**Key-words:** *shape memory, partial recovery, recovery load*

### 1. INTRODUCTION

The use of shape memory alloys in different applications in modern engineering is expected to increase significantly in the next decade, Dunn (2000). Advances in thin film technologies and fabrication processes are promoting the fabrication of micro-sensors and micro-actuators to integrate light robust control systems. The reduced dimensions of these systems requires a precise determination of the response characteristics of shape memory material under the most variable thermomechanical operating conditions in order to recommend its use for a given micro-system.

Allied to their high mechanical resistance, NiTi shape memory wires are being used in various prototypes due to the alloy exceptional resistance to corrosion in aggressive atmospheres. In a recent paper, El-Sharawy and Viana (2001), reported the results of a first investigation of the effect of a sequence of thermomechanical loading on the behavior of a near equiatomic NiTi wire. The authors showed that, depending on the test temperature, form recovery may be inhibited if wire deformation is carried on beyond the load plateau level. Also, wire loading and recovery characteristics are shown to be insensitive to loading rates of 1mm/min or lower.

Based on these preliminary results, a number of research directions are being sought, including the effect of variable thermomechanical loading parameters. In this paper, the effect of *reloading* at higher temperature on shape recovery at *lower temperatures* will be analyzed as well as the behavior during subsequent heating of the wire partially recovered at a relatively low temperature.

## 2. MATERIAL AND METHOD

A NiTi wire element, 0.75 mm in diameter, was loaded in tension according to the experimental sequence indicated in table 1. The experiments are a continuation of the original test sequence performed on the same wire element by El-Sharawy and Viana (2001), terminated by loading and total recovery at 60° C. The present test included loading the material in the austenite phase following the sequence of table 1. The tests spanned two months and included loading at 25°

Tabela 1. Thermomechanical testing sequence

EVENT	DESCRIPTION	OBSERVATIONS
1	Loading / unloading at 25° C	Same day
2	Repeated loading / unloading at 25° C	
3	Loading / unloading at 25° C	Consecutive tests 6 days later
4	2 <sup>nd</sup> loading / unloading at 25° C	
5	3 <sup>rd</sup> loading / unloading at 25° C, followed by heating from 25° C to 60° C	
6	Loading / unloading at 25° C	Consecutive tests 45 days later
7	Heating from 25° C to 70° C	
8	Reheating from 25° C to 70° C	
9	Loading / unloading at 60° C	Consecutive tests 6 days later
10	Loading / unloading at 40° C	
11	Loading / unloading at 30° C	
12	Loading / unloading at 25° C	
13	Heating from 30° C to 60° C	
14	Heating from 25° C to 70° C	
15	Heating from 25° C to 70° C	

C, subsequent heating through two heating ranges, loading at a higher temperature, reloading at the same lower temperature and subsequent reheating through the same temperature ranges. In all cases, loading proceeded at the rate of 1,0 mm per min in an EMIC testing machine. The wire was loaded while submerged in water in an acrylic tank and a digital thermostatic bath was used for temperature control. A circulating pump maintained a constant water level in the acrylic tank.

## 3. RESULTS AND DISCUSSION

Figure (1) shows the load profile of the wire stretched along 3 mm of deformation. The wire was extended beyond the range of the load plateau indicating a complete austenite → martensite deformation induced transformation. Unloading followed down to the initial starting load, though was not recorded in this initial test. The second consecutive loading curve is shown in Fig. (2). The test shows a linear load increase, without a load plateau, and the wire was unloaded after a shorter deformation to avoid component fracture. The absence of a load plateau indicates no phase transformation. Loading under the same conditions was repeated 6 days later and the linear plateau-less load behavior was confirmed for three consecutive tests as shown in Fig. (3) and Fig. (4).

These results confirm the inhibition of phase transformation on consecutive loading at 25° C. This is attributed to the level of residual strain sustained by the wire upon unloading after the first loading experiment, Fig. (1). This shows that, in this case, form recovery takes place only partially. The increase in the level of residual strain is probably insignificant after subsequent testing due to the linear, plateau-less behavior which permits a closed loop on unloading, Fig. (3) and Fig. (4).

Since shape memory behavior may be “tailored” during thermomechanical “training” sequences, Otsuka and Wayman (1998), the authors sought repeating the loading experiment at a

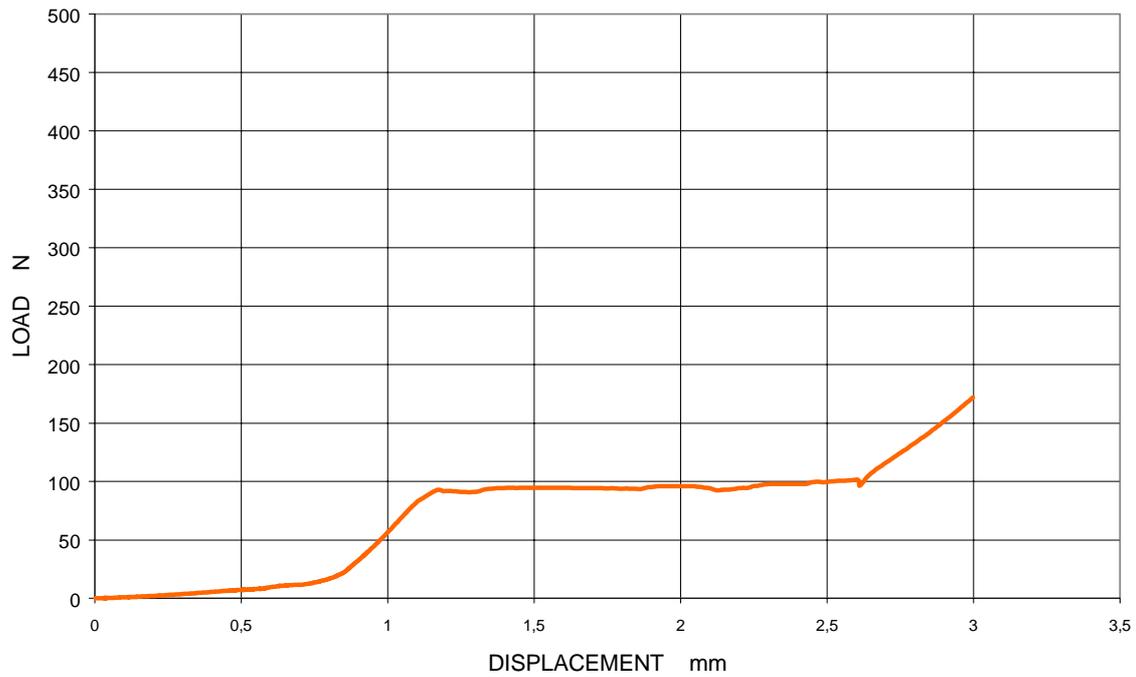


Figure 1. Wire behavior after 3.0 mm of deformation

higher temperature, 60° C, in an attempt to recover the pseudo-elastic behavior at 25° C and the associated shape recovery capacity of the material. Furthermore, the wire was subjected to a number of heating sequences in order to evaluate the level and the effect of residual strain, after partial form recovery, on the wire response on subsequent thermomechanical loading. Heating was performed while the wire is maintained attached to the grips under a minimum load. The first heating experiment was performed at the end of the third consecutive loading, as indicated by the wire response after unloading, Fig. (4). The wire was heated from 25° C to 60° C at a rate of 0,05° C

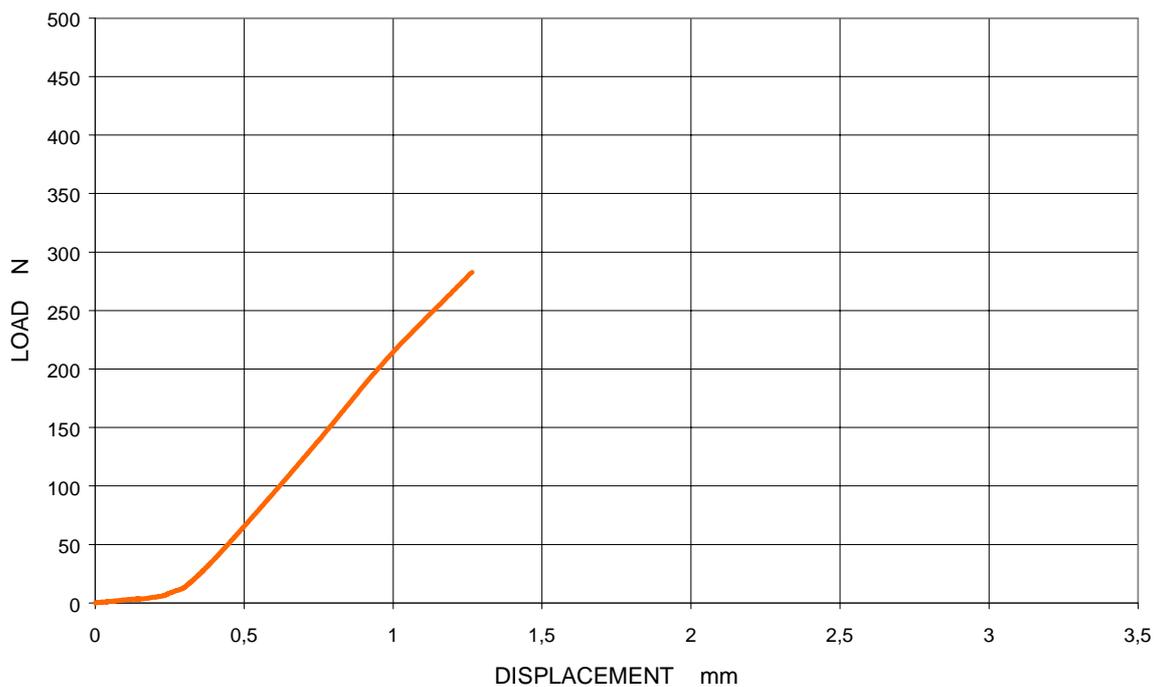


Figure 2. Second consecutive loading at 25° C - same day

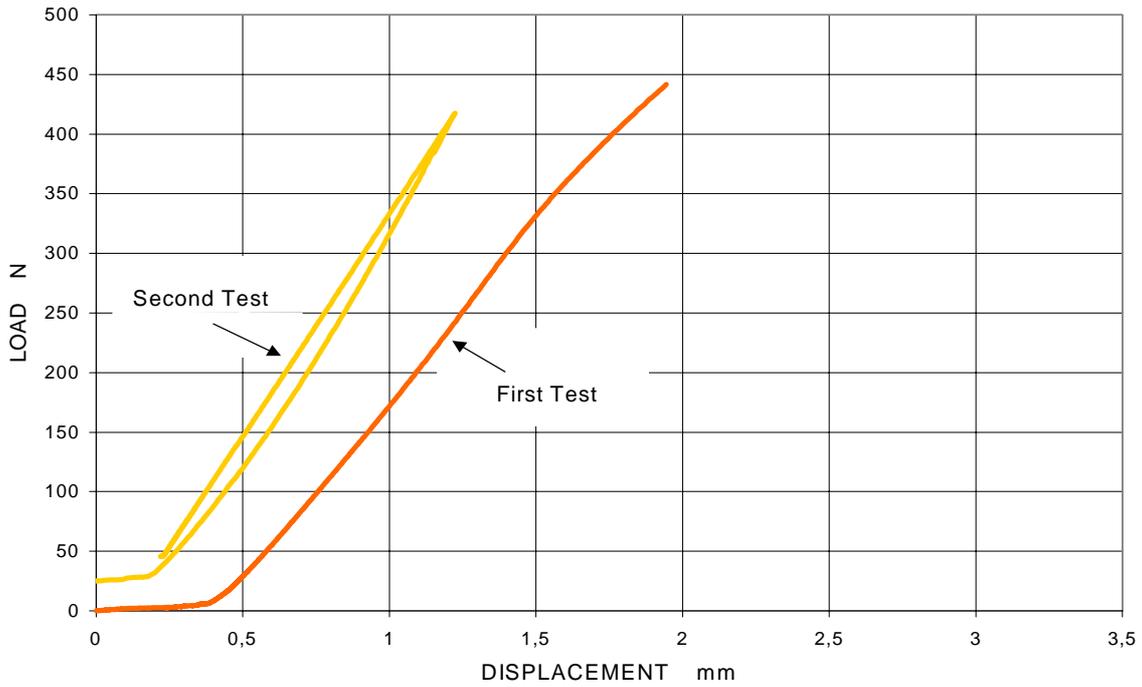


Figure 3. Consecutive loading at 25° C - 6 days later

per second, and maintained for 9 minutes at maximum temperature. Figure (5) shows that the wire immediately reacts to heating by developing a continuously increasing tensile recovery load, stabilizing at a plateau during permanence at maximum temperature. This indicates that the wire is highly unstable under the effect of the residual strain, immediately reacting in the direction to restore the original unstrained length as the temperature is increased, sustaining as a result a continuously increasing load. A continuously increasing ‘recovery load’ indicates that the

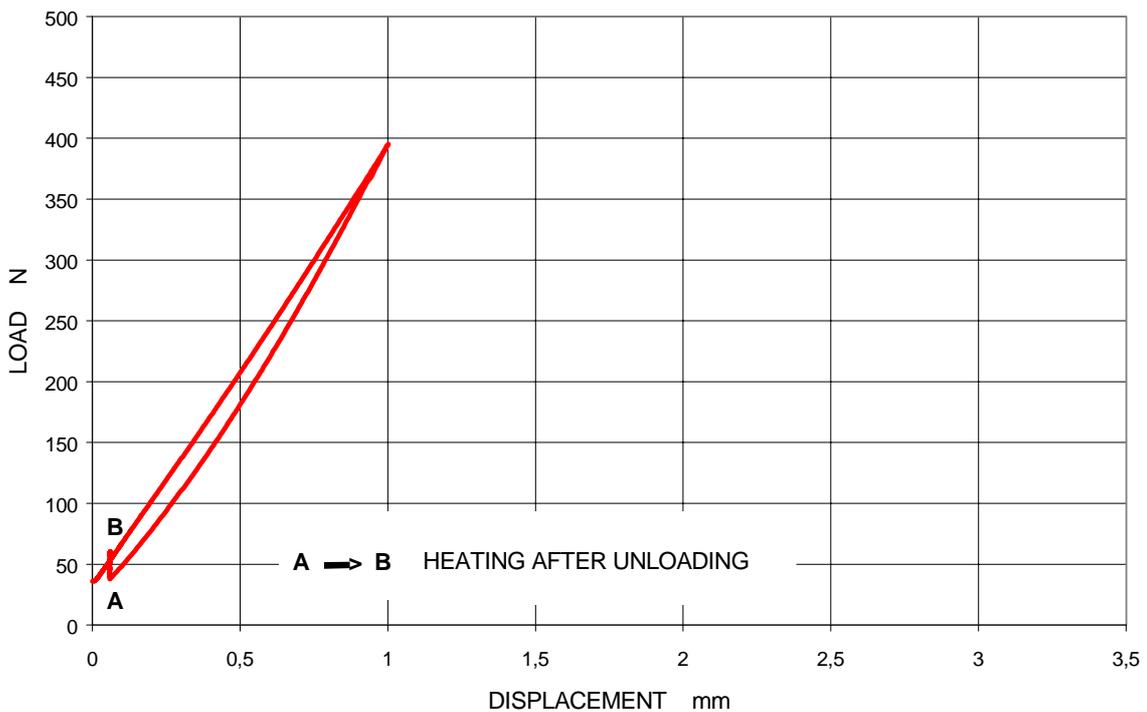


Figure 4. Third consecutive loading at 25° C - same day

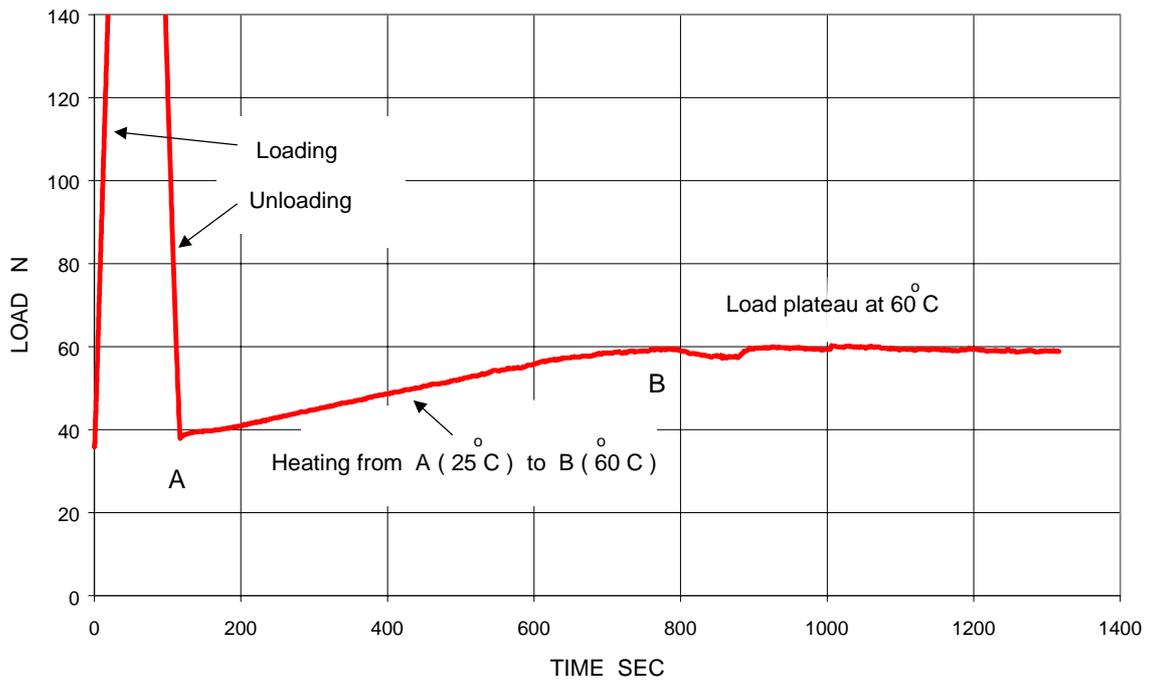


Figure 5. Development of recovery stress on heating after unloading of the third consecutive test

heating temperature range is probably not sufficient to eliminate all the residual strain. This hypothesis was confirmed as loading was resumed. Figure (6) shows that, 45 days later, the wire load response continues to be linear with no tendency for a plateau formation. It should be mentioned at this stage that each time a set of experiments is concluded on a given day, the assembly containing the acrylic tank and the wire attached to the grips was removed from the testing EMIC machine as an integrated unit. In this manner, the wire remained coupled at all times to two light gripping elements, permitting quick repositioning on test resumption. Also, while off the

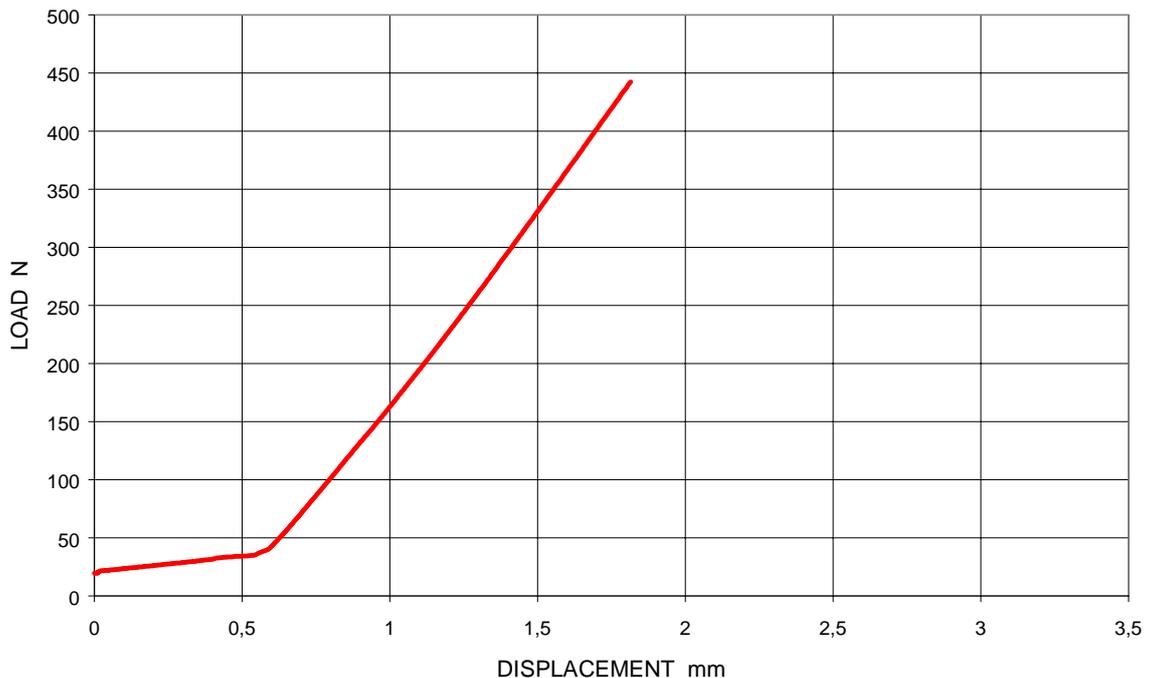


Figure 6. Wire loading behavior at 25°C - 45 days later

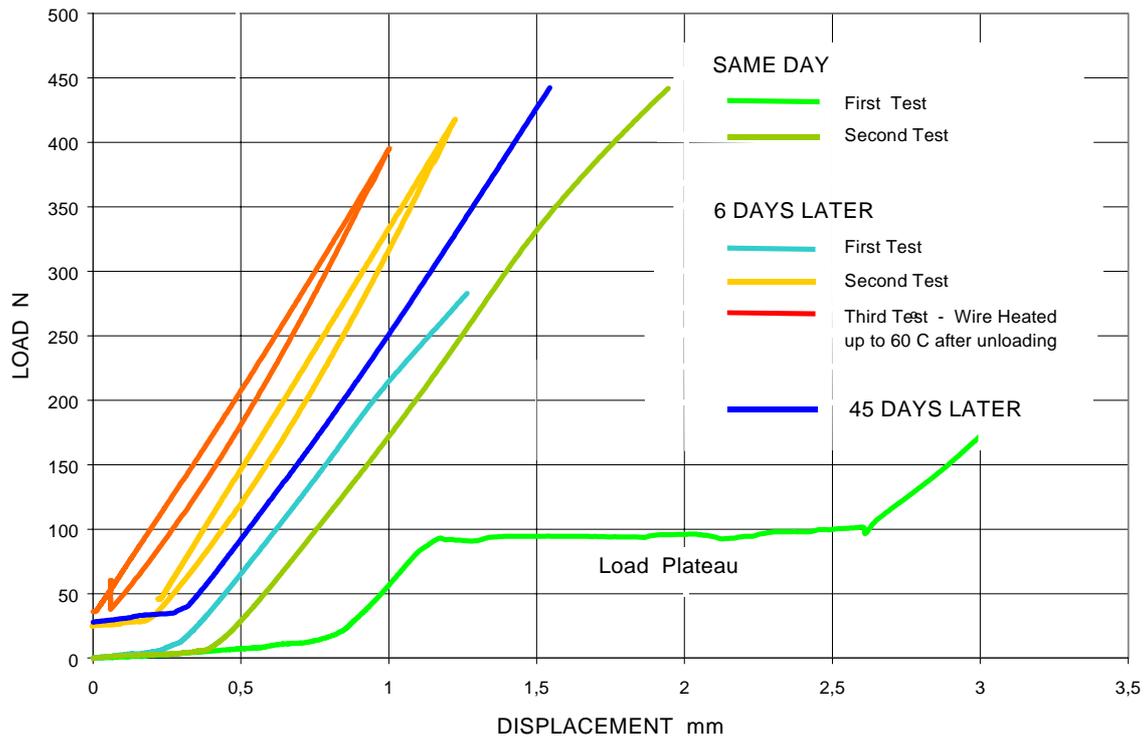


Figure 7. Loading history at 25° C

machine, the wire was only under the effect of the small load of only one of the grips (~50 gf). Therefore, the wire was practically free to restore its original length while off the machine. However, the loading history in Fig. (7) shows that, after 45 days, loading proceeded up to the same high level as the second loading at the start of testing. That is, the residual strain condition was not significantly altered during the off period. This strongly indicated phase transformation and associated form recovery was definitely inhibited at that stage of loading at 25° C.

The wire was next heated in a sequences of two tests from 25° C to 70° C in order to analyze the level of the residual strain. Figure (8) shows that recovery during the first heating test is slow up to approximately 1000 seconds (corresponding to a temperature > 60° C). The recovery load increases rapidly afterwards and levels off as before at a plateau during the period of permanence at maximum temperature. The wire was next cooled down back to 25° C, at approximately the same heating rate. At the end of cooling, the wire sustained a higher load (~ 35 N) as compared to the load at the beginning of heating (~ 17 N). As shown in Fig. (8), the sustained load was adjusted down to the initial level before the start of the second heating experiment. Again, the 'recovery load' increased initially at a slow rate, and at significantly higher rate above 60° C. The 'recovery load' profile was repeated, leveling off at the same load plateau sustained by the wire at maximum temperature at the end of the first heating experiment.

Figure (9) shows a comparison of the heating test results. It is clearly observed that, judging by the higher 'recovery load', more strain recovery is obtained as the maximum temperature is increased. Also, the difference in the observed recovery rates, as indicated by the different curve profiles, is a function of the load level sustained at the beginning of heating; it is assumed that the residual strain sustained by the wire has not increased between the first and the third heating tests (event 5 to 8 in table 1). In other words, the strain recovery rate is a function of a given residual strain and the initially sustained load.

Moreover, the observation of a nearly repeatable 'recovery load' profiles for the two consecutive heating tests, Fig. (8), including an equal load plateau level, indicates that the residual strain state is effectively restored after cooling. Therefore, it may be concluded that heating encourages only a temporary strain recovery.

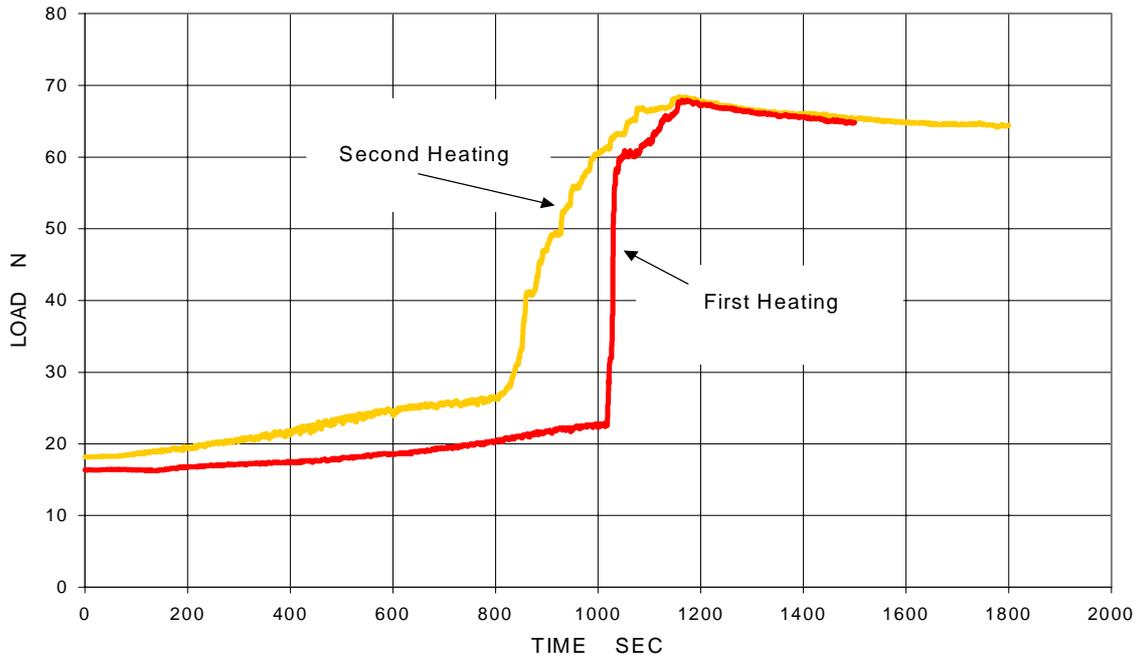


Figure 8. Wire load profile during heating from 25° C to 70° C

The above results show that, for the given shape memory material, a heating input is probably insufficient to restore the capacity of phase transformation and associated shape recovery at 25° C. Consequently, loading at a higher temperature was sought as an objective thermomechanical option. Figure (10) shows that total shape recovery is attained after unloading at 60° C. In order to analyze the effect of total shape recovery on subsequent loading, the wire was consecutively loaded at intermediate temperatures; 40° C and 30° C, and finally at 25° C. Figure (11) shows that partial shape recovery takes place at the lower temperatures. As expected, all curve profiles are similar, the load increasing initially slowly along the same path, though through different extensions. Also, the

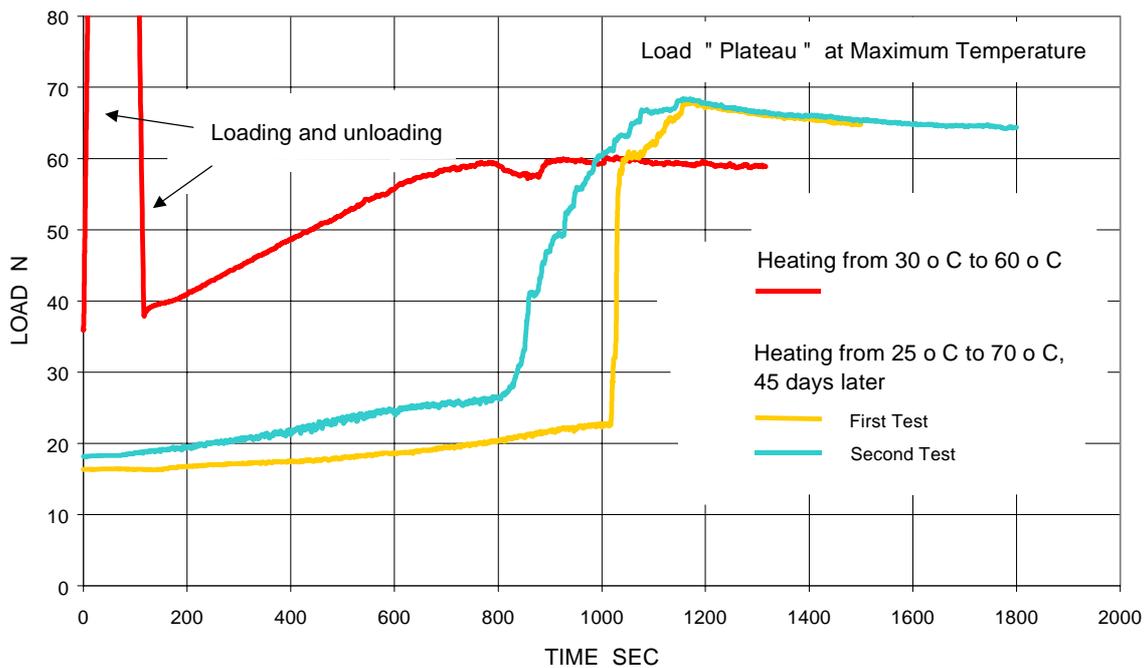


Figure 9. Combined wire recovery load during heating

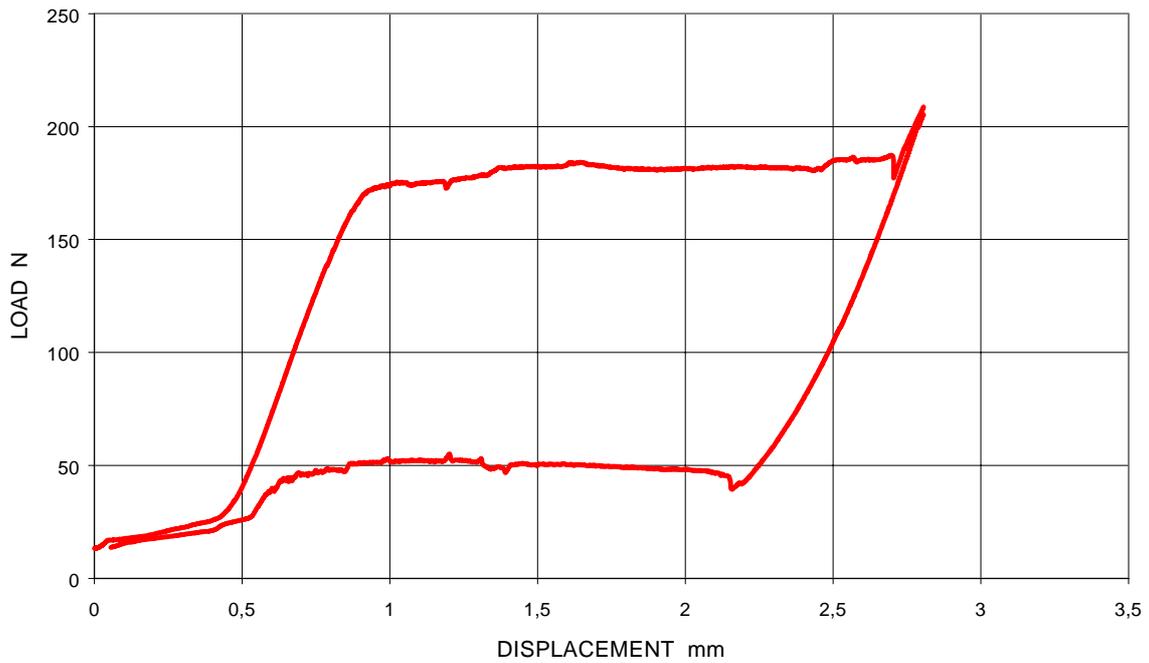


Figure 10. Loading at 60° C

sharp load increase follows along parallel paths. The transformation load plateau decreases as the loading temperature decreases. This is coherent since a higher austenite  $\rightarrow$  martensite transformation load plateau is required as the test temperature,  $T_t$ , and consequently the difference  $T_t - M_s$ , is increased. The partial form recovery on consecutive loading at 40° C, 30° C and at 25° C is a strong indication that recovery was also partial after first loading at 25° C, where the unloading curve was not recorded. Therefore, the above analyses of wire behavior based on the presence of residual strain is justified. Figure 12 shows the expected similarity between the loading curves at 25° C; at the beginning of testing and after reloading at 60° C. Nevertheless, the comparison

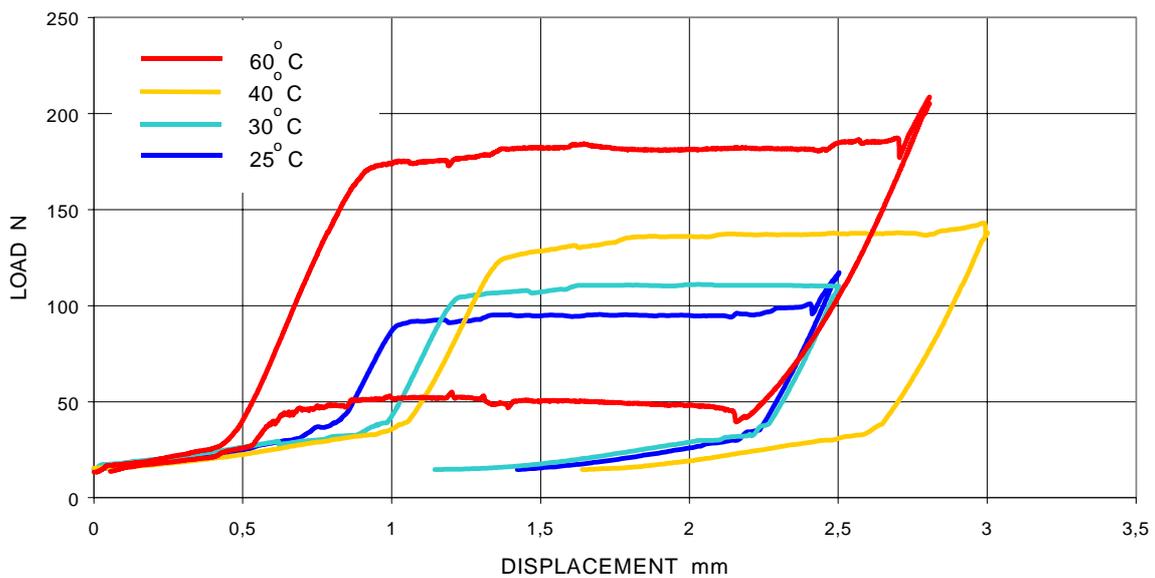


Figure 11. Total and partial form recovery at different temperatures

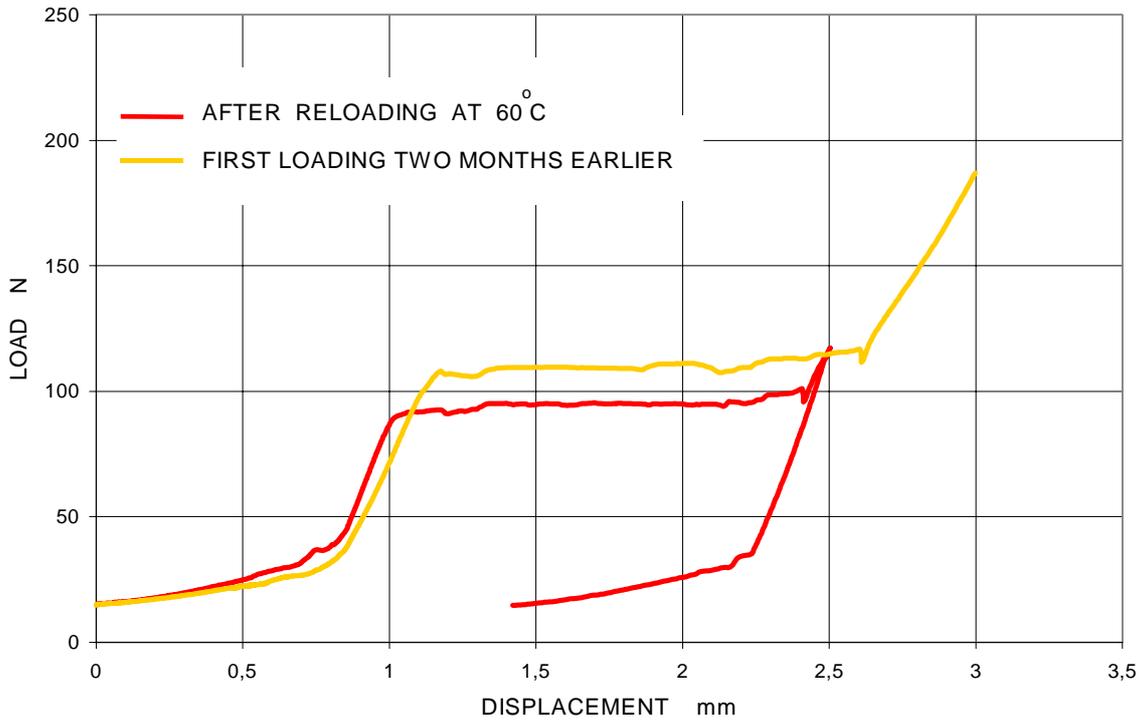


Figure 12. Comparison of wire loading profiles at 25° C

shows that, after reloading at 60° C, the loading curve at 25° C returns a lower and shorter load plateau, an alterations of the wire characteristics due to repeated thermomechanical loading. That is, phase transformation takes place at a lower load during a shorter period. The shorter period indicates either a faster or an incomplete phase transformation.

In order to analyze the level of residual strain after the second phase transformation and partial recovery at 25° C, the wire was heated through the ranges described by events 13 to 15 in table 1. Figure 13 shows that, on heating to 60° C (red curve), the level of strain recovery ( indicated

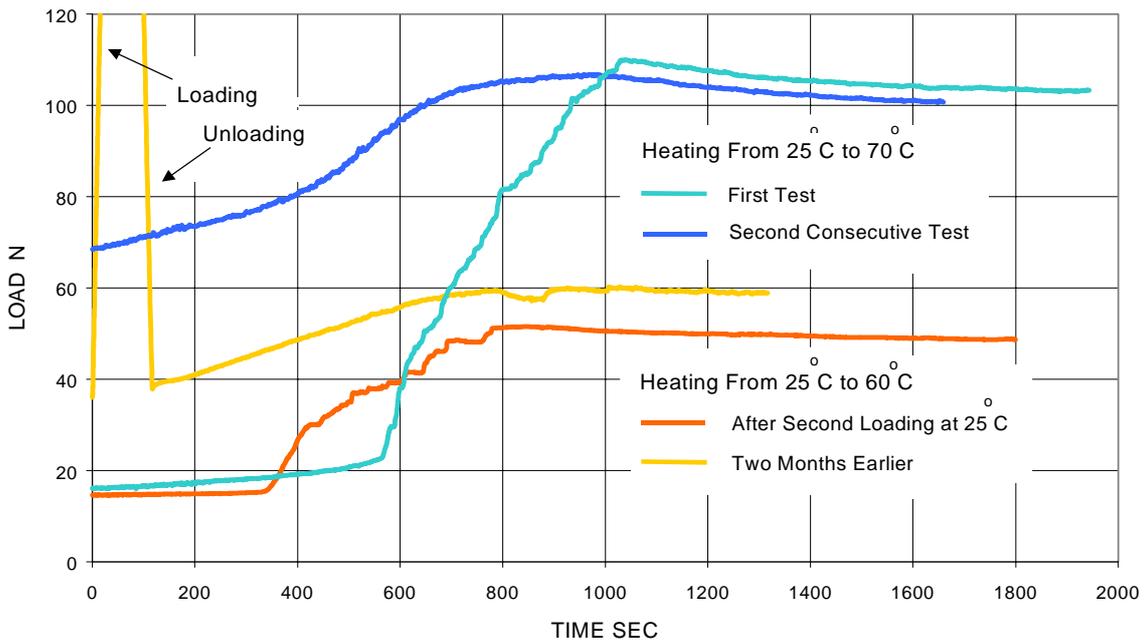


Figure 13. Wire recovery load along two heating ranges

by the load plateau ) is comparable to the previous recovery obtained during the first heating test (orange curve). However, consequent heating to 70° C shows a significant increase in strain recovery. Again, it is observed that strain recovery is *temporary*, judged by the repeated load plateau obtained at maximum temperature ( green and blue curves ).

Figure (14) compares the recovery load profiles for the heating tests up to 70° C. The wire load before heating was adjusted down to the same level for three of the four tests. The initial load before the fourth test (green curve) is significantly higher, and corresponds to the unadjusted residual load on cooling down after the third heating test. In spite of the significant difference the third and fourth recovery loads converge up to the same plateau level. these results show that strain recovery on heating is definitely a function of the residual strain and the maximum heating temperature.

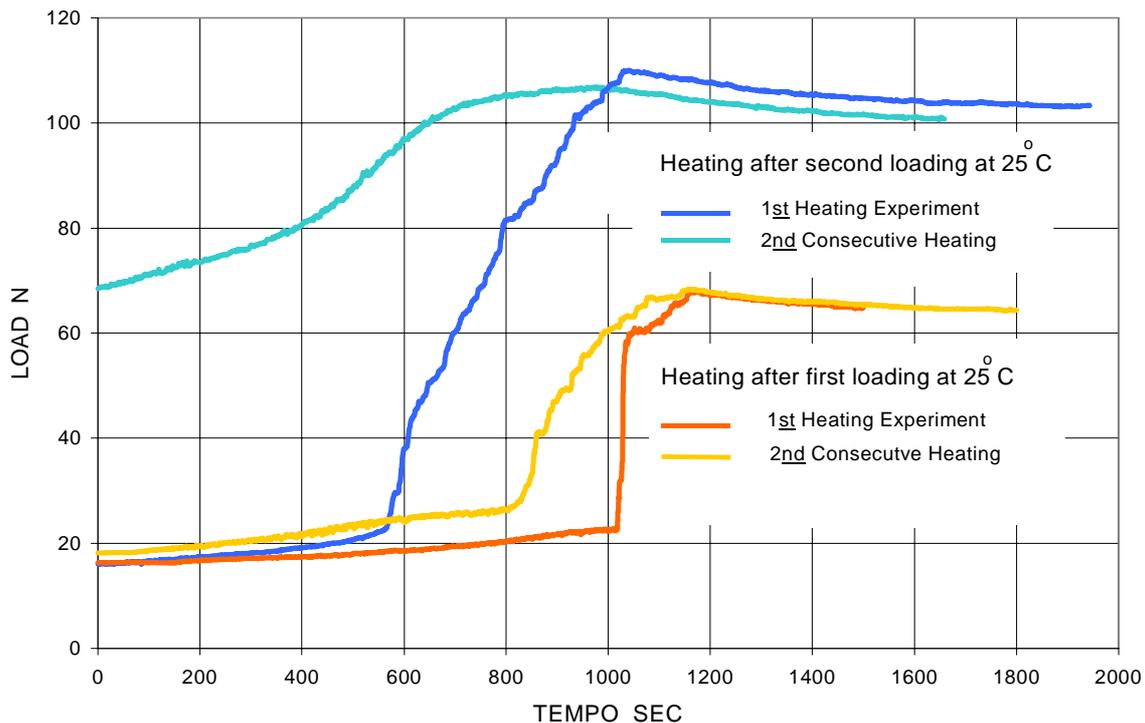


Figure 14. Combined wire reaction stress during heating from 25 o C to 70 o C

#### 4. CONCLUSIONS

Thermomechanical loading of a NiTi wire in the austenite temperature range shows that the capacity of phase transformation and related shape recovery are interrupted on repeated loading at 25° C, at which shape recovery is partial.

Partial shape recovery at 25° C is restored after reloading at a higher temperature, 60° C, at which shape recovery is complete.

Temporary strain recovery is obtained as long as the heating temperature is maintained

#### 5. REFERENCES

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