J. Phys. IV France **11** (2001)

Stress/temperature phase diagrams as a tool for shape memory alloy selection and processing

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One of the difficulties of applying shape memory alloys to real applications is dealing with how applied stress affects the transformation temperatures and phase sequence. This paper presents stress-temperature phase transformation diagrams of the type described by Todoroki, Tamura and Suzuki [1]. The data employed to construct these diagrams relates to a previous study of binary NiTi alloys that have varied alloy compositions, prior cold work and heat treatment temperatures [2]. The diagrams presented in this paper graphically display how phase sequence as well as transformation temperatures vary with processing and alloying parameters. In addition, it is shown that the stress-temperature diagrams change after repeated thermal transformations against applied stress. Increasing M_s temperatures and decreasing A_s temperatures result in narrower thermal hysteresis and loss of the intermediate R-phase.

1. INTRODUCTION

This work builds on a previous study on the stability of the memory effect in NiTi shape memory alloys when cycled against constant stress [2]. In the previous study, permanent strains and degradation of recoverable memory was studied on 50.26at%Ni-Ti alloys. The experiments were designed so that the relevant significance of factors such as cold work and heat treatment could be assessed.

The trial carried out in the following paper uses a similar design of experiments. However, this time the purpose is to analyse changes in transformation temperature during constant stress cycling. Stress verses temperature plots are constructed to:-

a) Show graphically how the transformation temperatures drift with cycling.
b) Assist in the selection of alloy type and the required processing procedure.

2. PROCEDURE

Five variables were identified as being of particular relevance to the thermo-mechanical fatigue of NiTi alloys. Upper and lower values were chosen for each variable and these are shown below in Table 1.

Table 1: Variable Levels

Level	Heat Treatment Temperature °C	Applied Stress MPa	Prior Cold Work %	Alloy Type at% Ti
Upper	500	165	30	50.12
Lower	400	85	6	49.74

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jp4:2001884

Thermal transformation cycling was carried out using resistance heating of the wire. A current applied across the ends of the wire was gradually increased for the heating run and decreased for the cooling run. Different weights were attached on the free end to provide a constant stress during cycling.

A thin foil, K-type thermocouple was attached directly to the wire to monitor temperature whilst a laser displacement measuring device was employed to measure strain. Temperature and displacement data was sent directly to a PC running LabView® control software.

Transformation temperatures were measured via tangential lines drawn against the hysteresis profile.

3. RESULTS

The measured M_s and A_s transformation temperatures are plotted against applied stress level for cycles number 1 and 500 in figures 2 to 9. The M_s and A_s temperatures at zero stress (as measured by DSC) are also included to complete the cycle 1 curves. Cycling at zero stress was not carried out and therefore the transformation temperatures after thermal cycling are only plotted for the 85MPa and 165MPa tests. Where appropriate the R-phase peak temperature has been plotted.

The curve at cycle 1, where the alloy has the same starting microstructure is indicative of a Clausius-Clapeyron type relationship [3]. Strictly speaking, the curves plotted at cycle 500 do not show true Clausius-Clapeyron behaviour because the alloys have been cycled at different stresses to obtain the data points and therefore are likely to have different microstructures.

The results are plotted in Figure 2 to Figure 9 such that each graph represents a particular alloy (Ni rich or Ti rich), with a fixed amount of prior cold work (6% or 30%) and a fixed heat treatment temperature (400°C or 500°C).

Due to high residual strains the transformation temperatures of the samples shown in Figures 8 and 9 could only be measured up to cycle number 30.

3.1 Summary of Results

1. In general, the alloys tended to show an increase in the M_s temperature with constant stress cycling and a decrease in A_s.

2. In all tests alloys that contained low prior cold work, were heat treated at high temperatures and were subjected to higher applied stress levels tended to show the greatest drift of transformation temperatures.

3. Changes in transformation temperatures were such that the width of hysteresis decreased during cycling. In addition the slope of the hysteresis curve tended to increase. These changes are shown in Figure 1. Again, these changes tended to be greatest in the samples with low prior cold work, heat treated at high temperatures and cycled against the higher stress level.

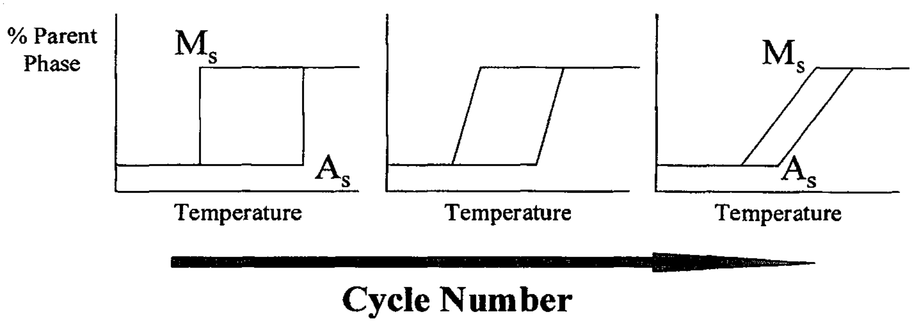

Figure 1: Observed Changes In Hysteresis Profiles

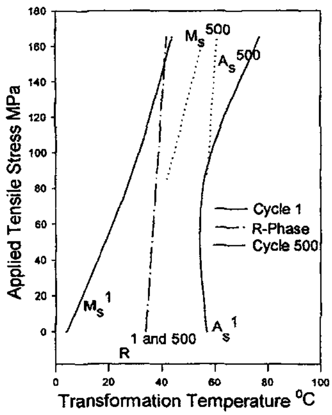

Figure 2: 50.26at%Ni-Ti, 30% CW, 500°C HT

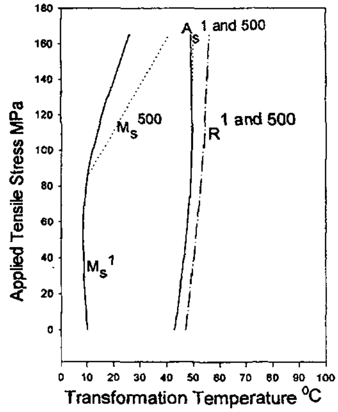

Figure 3: 50.26at%Ni-Ti, 30% CW, 400°C HT

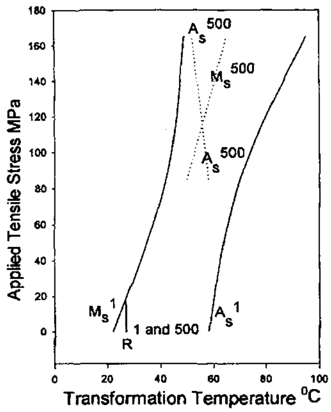

Figure 4: 50.26at%Ni-Ti, 6% CW, 500°C HT

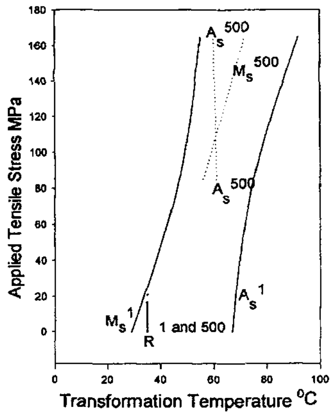

Figure 5: 50.26at%Ni-Ti, 6% CW, 400°C HT

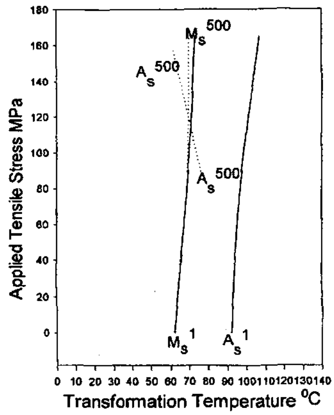

Figure 6: 49.88at%Ni-Ti, 30% CW, 500°C HT

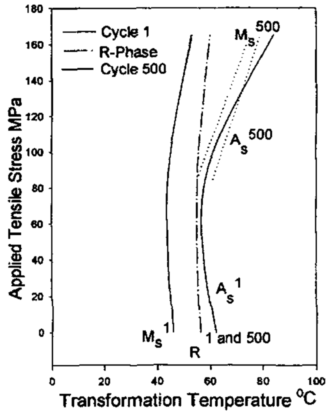

Figure 7: 49.88at%Ni-Ti, 30% CW, 400°C HT

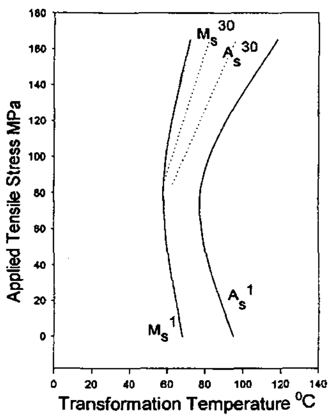

Figure 8: 49.88at%Ni-Ti, 6% CW, 500°C HT

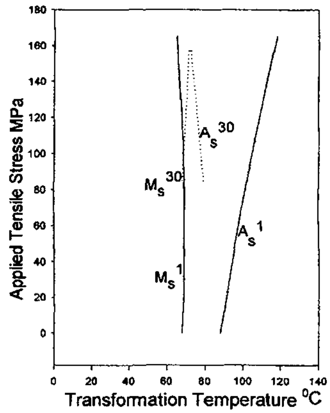

Figure 9: 49.88at%Ni-Ti, 6% CW, 400°C HT

4. In the 30% cold worked, Ti rich alloys (Figures 6 and 7), the A_s temperatures only decreased in the samples heat-treated at 500°C. The A_s remained virtually constant for those heat-treated at 400°C.

5. In some of the alloys, the A_s at 165MPa decreased at a greater rate than the A_s at 85MPa. This resulted in a higher measured M_s than A_s.

6. The R-phase transformation was only observed in alloys where the M_s temperature tended to be below approximately 30°C - 40°C in the Ni rich alloys and below approximately 50°C in the Ti rich alloy.

7. Although the R-phase temperatures remained constant during cycling a gradual increase of the M_s temperature meant that the R-phase was effectively suppressed in certain alloys (i.e. Figures 2 and 7)

4. DISCUSSION

4.1 Thermo-mechanical effects

The increase of M_s and decrease of A_s observed in many of the alloys results in a narrowing of the hysteresis width and increase of slope (i.e. temperature interval between the start and finish of transformation). It is considered that hysteresis width is dependant upon internal friction [4] - the lower the internal friction the smaller the hysteresis. Slope of the hysteresis is considered to be dependant upon elastic energy [4] – the greater the elastic energy the greater the slope. Therefore the results presented in this paper imply that cycling against constant stress reduces internal friction and increases elastic energy.

As both effects depend upon dislocation density and distribution it is logical that those samples which are most susceptible to changes in dislocation density should show the greatest changes in hysteresis and transformation temperatures. This explains why the greatest changes in transformation temperatures were observed in those alloys that had little prior cold work, high heat treatment temperatures and were cycled against the comparatively high stress level. In a previous study it was also these alloys that were found to show the greatest permanent shape strains during constant stress fatigue studies [2].

The rate of the M_s and A_s drift was found to be greatest in the early cycles and by cycle 500 generally became quite stable. These results agree with the hypotheses of other authors who also attribute cycling effects to the generation and saturation of dislocations [5][6][7].

4.2 Two-way training

These results confirm that cycling against constant stress results in changes to the internal microstrcuture and transformation behaviour of the alloy. As cycling against constant stress is a recognised method of inducing two-way memory [8] these results also have implications for two-way memory design. The two-way effect is a result of internal stress fields thought to result from domains of high dislocation densities that in turn exert forces on the transforming martensite plates and cause predominant variants to form [9].

The results presented in this paper confirm that training an alloy for the two-way effect may cause changes to the transformation temperatures. If a two-way actuator is designed to operate over a particular temperature window or transform at an exact temperature it is not enough to take cycle 1 temperature data only or worse still the manufacturers ingot data.

4.3 Alloy selection

The stress temperature diagrams can be used for alloy selection of both 'one off' actuators and repeatable multi-cycle actuators. It can be seen in Figures 4 and 5 how applying a stress to an alloy that apparently shows perfect R-phase properties under zero stress in the DSC may in fact lose this transformation due to the increase of the M_s following the Claussius-Clapeyron law.

Equally, the R-phase transformation may be lost due to the gradual increase of M_s during thermal/stress cycling. For instance the alloy cycled at 85MPa in Figure 2 showed R-phase transformation and martensite transformation during early cycles. However, as the cycle number increased the M_s increased significantly and eventually the R-phase was lost.

In summary, the results show that to attain a stable R-phase transformation the M_s must be depressed. This is achieved by retaining a high amount of cold work, i.e. heat treating an alloy that has high prior cold work at a low temperature and by increasing the Ni content. This is also in agreement with other authors [10]. Even in these alloys a high level of applied stress may destroy the R-phase. In Figure 3 the M_s at 165MPa increased significantly during cycling and by cycle number 500 was just 8°C below the R-phase peak temperature.

The stress-temperature diagrams also demonstrate how transformation temperatures may be 'fine-tuned' by stress/temperature cycling. Of particular significance from this point of view is the ability to reduce the hysteresis width and thus reduce the temperature window over which the alloy operates.

5. CONCLUSIONS

- The changes in transformation temperatures and hysteresis observed during the experiments described in this paper are due to decreasing internal friction and increasing elastic energy.

- The samples that showed the most stable transformation temperatures were those with high prior cold work heat-treated at comparatively low temperature.

- Converting a one-way memory to a two-way memory using constant stress cycling causes the transformation temperatures to change significantly.

- R-phase is most stable in those alloys that are Ni rich, have high prior cold work and are heat-treated at comparatively low temperatures.

- The R-phase transformation may be lost in those alloys where the M_s increases to such an extent during cycling that it suppresses the R-phase.

- The hysteresis width may be reduced after initial processing by thermal cycling at constant stress.

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