

## Thermal and mechanical fatigue of shape memory alloys

M. Sade<sup>1</sup>, E. Hornbogen, Institut für Werkstoffe, Ruhr Universität Bochum, FRG.

### Introduction

Different modes of fatigue can be induced in shape memory alloys. Thermal fatigue is defined as due to cyclic temperature changes in the range for which austenite ( $\beta$ ) is stable:  $T_{max} > A_f$ , down to temperatures at which the transformation is complete  $T_{min} < M_f$ . Several modes of mechanical fatigue are possible. They include fatigue of stable austenite ( $T > A_f$ ), of stable martensite ( $T < M_f$ ), during pseudoelastic cycles ( $M_d > T$ ,  $M_d$  = highest temperature to which martensite can be stress induced). Each of the mentioned modes provides different microplasticity mechanisms (1). Several intermediate thermomechanical fatigue treatments are also possible. An example is the thermal cycling through the martensitic transformation, under a simultaneous application of load.

Fatigue of single and polycrystalline Cu base shape memory alloys has been studied and the results can be found in the literature (2,12). In the studies on fatigue in single crystals of Cu based alloys the dependence of the number of cycles to fracture on several parameters, i.e. applied stress, axis orientation, test temperature, etc. (3,4,5,9,10) and their relationship to structural changes have been analysed (13). It has been shown that due to pseudoelastic cycling of Cu-Zn-Al alloys the density of dislocations which form crystallographic arrangements increases in the interior, that martensite is retained, and that at the surface slip steps, intrusions, extrusions and microcracks develop.

Most of the works however, have been performed in polycrystals (6,7,8). Grain boundaries in bcc long range ordered shape memory alloys provide sites of severe incompatibility because of the high elastic anisotropy. They are often extremely brittle, for example in the intermetallic compound  $\beta$ -CuZn (14). Fatigue of polycrystals of shape memory alloys has predominantly shown the presence of intercrystalline fracture. For fatigue crack growth grain boundaries can be sites for impediment or provide an easy intercrystalline path (15,16).

It is the purpose of this paper to summarize some results obtained with single crystals, and discuss their relevance for polycrystalline materials. In this paper emphasis will be put on the relationship between the different modes of fatigue in the shape memory alloys. It will be discussed to what extent single crystal studies can give information also on the behavior of polycrystals, and on what factors this depends. For this reason the experimental procedures and results of existing data will be summarized and some new information will be presented, all for

<sup>1</sup>Humboldt-Fellow, on leave from Centro Atómico Bariloche, Argentina

Cu-Zn and Cu-Zn-Al alloys. Here  $\beta$  will be used to designate the austenite and M de martensite, indistinctly for CuZn and CuZnAl alloys. M represents the 9R structure in Cu-Zn alloys and 18R in Cu-Zn-Al alloys.

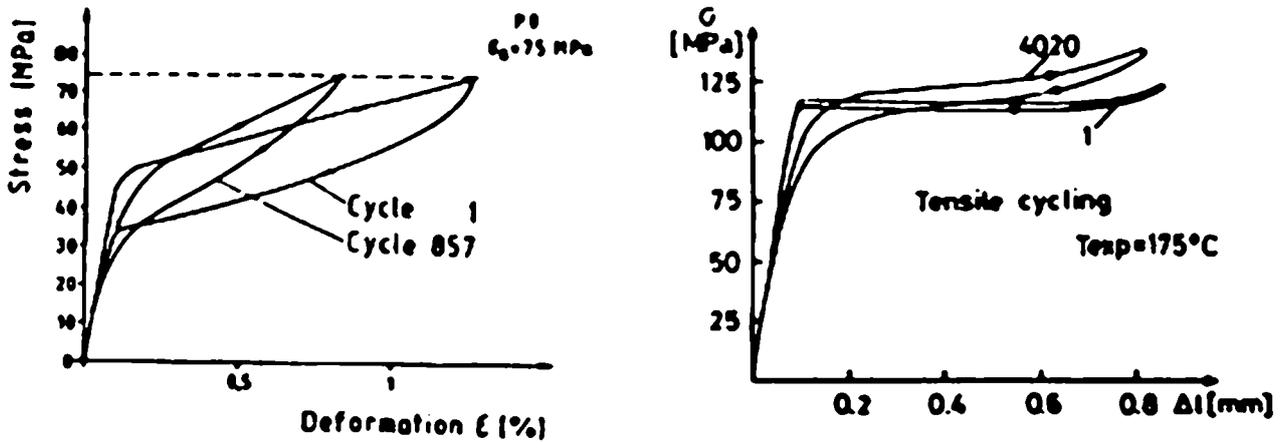
### Experimental procedure

The experimental procedure has been explained in previous reports (1,3). Only a short summary is given here. The single crystals were grown by the Bridgman technique. Samples were annealed for 2 hours at 800 C and then air cooled. Their surfaces were mechanically (600 grade paper) and electrolytically polished. Mechanical cycling was performed in closed loops at different test temperatures above  $M_s$ . The maximum deformation was kept constant during the cycling of the single crystal specimens. Light and electron microscopy (SEM and TEM) were used to study the defect structures after cycling. Thermal cycling under load was performed in a specially designed temperature chamber (17). A Cu-Zn-Al alloy of  $M_s = 10^\circ\text{C}$  (Cu-24.26 at % Zn-3.32 at % Al) was chosen for experiments with polycrystalline specimens. The samples were annealed at 800 C for 2 hours and water quenched.

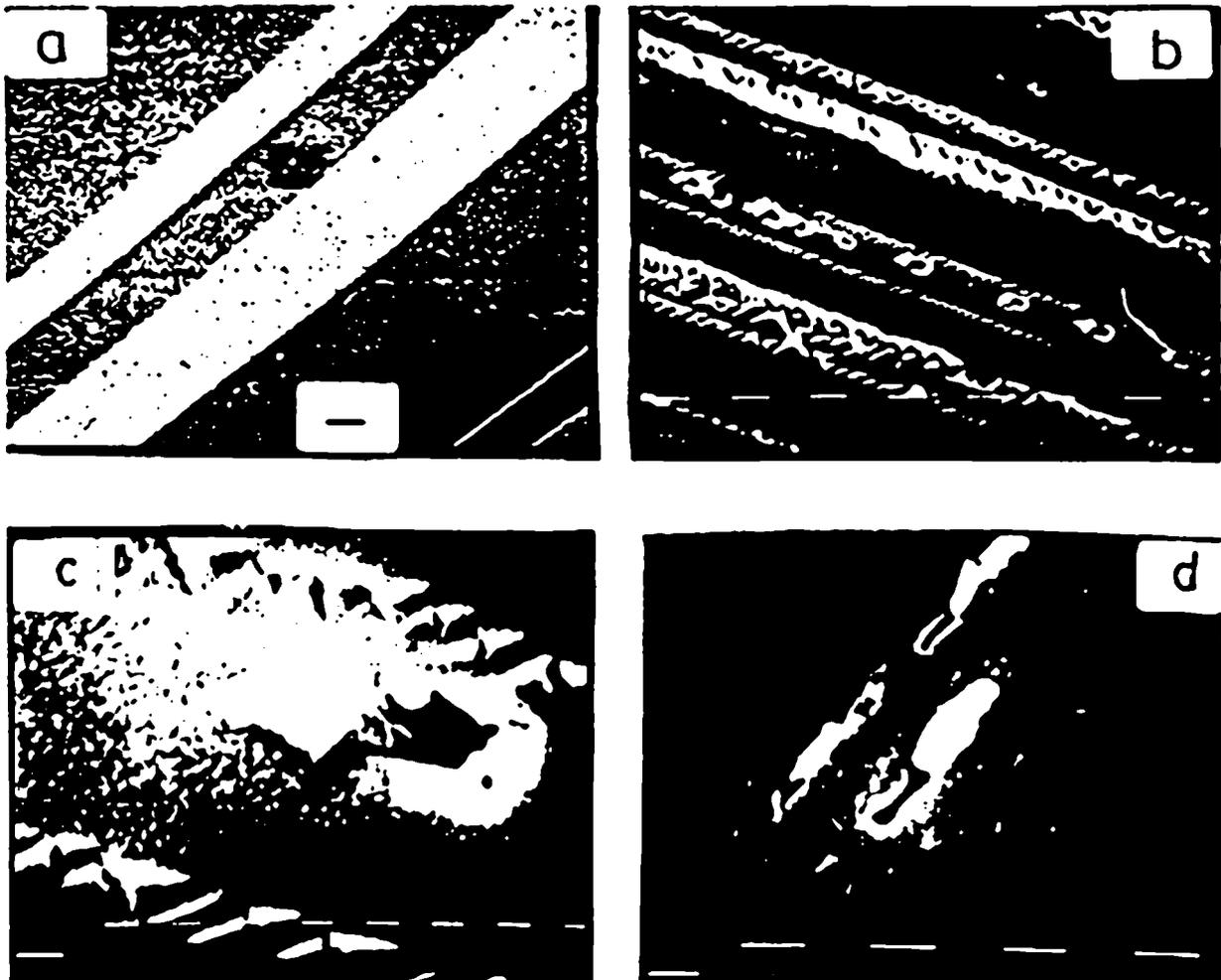
### Experimental results

Figure 1a shows stress strain curves for a polycrystalline sample after several pseudoelastic cycles, for a maximum stress of 75 MPa. The maximum stress was kept constant during cycling. A strong change in the slope of the stress-strain curve can be noted, which implies that a constant maximum deformation during cycling (as used in single crystals) would lead after a low number of cycles to high stresses and fracture. However the experimental conditions were controlled in order to allow a maximum plastic deformation of approximately 1%. A higher amount of plastic deformation led to intercrystalline fracture at a low number of cycles. A softening associated with the start of the transformation and a hardening during the progression of the transformation is observed. Consequences of this cycling on the temperature of transformation can be found in Ref. (18). The evolution of stress strain curves during pseudoelastic cycling under tensile load for a single crystal is presented in fig. 1b. An increase in the hysteresis, a smoother onset of the  $\beta$ -M transformation and a hardening during the transformation are present after cycling.

The most detailed results have been obtained for a single crystal of a Cu-15.14at%Zn-16.43at.%Al alloy, of  $M_s = -5^\circ$ , samples of which were pseudoelastically cycled at constant temperature. Figure 2 shows one of the samples under tensile stress and a deformation corresponding to 50% of the pseudoelastic transformation (2a: before cycling, b,c and d: after pseudoelastic cycling). The boundary between martensitic bands and the austenite is clearly seen. Transmission electron microscopy studies as well as the evolution of stress-strain curves have been analysed previously (13,3). Dislocations form in the interior crystallographic arrangements which are elongated in the intersection between the martensitic habit plane and the basal plane of the martensite.

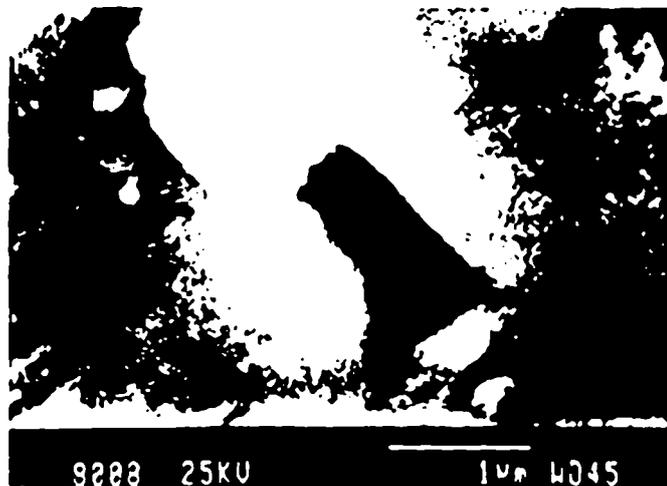


**Fig. 1:** Change in the course of  $\sigma$ - $\epsilon$  curves with the number of pseudoelastic cycles. a) polycrystalline sample, maximum stress = 75 MPa, b) single crystal specimen,  $M_s = -20$ .



**Fig.2:** S.E.M. analysis of surface defects after pseudoelastic cycling of single crystals (a,b,c in situ observation under load). Bar indicates 1  $\mu\text{m}$  ( $T_{exp} = 20^\circ$ ). a) partially transformed state, first cycle. b,c) Partially transformed condition after 3000 cycles. d) surface morphology after 3000 cycles as observed in the stress free condition.

The interaction of these defects with the surface give origin to slip steps. After additional cycling the steps grow in the surface and develop first extrusions-intrusions and finally micro-cracks which grow into the interior of the specimens. Characteristic is a banded structure which contains holes with tongue shaped extrusions (fig.2d). Light and scanning electron microscopy of the surface of polycrystalline samples have made visible defects of the same type of extrusions-intrusions as shown in fig.2 for single crystals. An example is presented in fig. 3. these defects are aligned parallel to the  $\{110\}_\beta$  planes which are parallel to the habit plane of the martensite. These habit plane defects are similar to those in single crystals and are thought to be responsible for the nucleation of microcracks which lead to fracture in single crystals, both in tension (3) and in compression (19). These experiments are an example of the close similarity with single crystalline surface defects provided, as will be shown, if strain incompatibilities at grain boundaries are kept low enough. Samples of the same alloy which were thermally cycled under load fractured at grain boundaries (18). However, when compared both kind of experiments it should be kept in mind that deformation is a free variable when the specimen transforms under constant load, and it reaches values higher than 5%. The amount of deformation is related to the stress concentration at grain boundaries.



**Fig.3:** Intrusion-extrusion at the surface of a polycrystalline specimen, after 758 pseudoelastic cycles.  $\sigma_{\max} = 25\text{MPa}$ ,  $\epsilon_{\max} = 0.8\%$ .  $T_{\text{exp}} = 20^\circ$ .

Strain compatibility problems at grain boundaries are the new feature if polycrystals are treated. Their consequences on fatigue crack growth and specimen life have been reported (1). It has been found that a large number of shear systems is induced near grain boundaries, and that intercrystalline fracture occurs associated with transcrystalline deformation, being the more pronounced the higher the test temperature is above  $M_s$ . It indicates that true plastic deformation and not transformation by self-accommodating groups is favouring grain boundary cracking.

There are at least two additional mechanisms which can favour transcrystalline deformation compared to intercrystalline fracture in polycrystals (1,17): a) Under cyclic loading in the martensitic state (i.e.  $T < M_f$ ), nucleation of cracks at various boundaries between martensite variants is favoured. Crack initiation has been found at variant boundaries with orientations normal to the external load after mechanical cycling at constant temperature  $T < M_f$  for an alloy of  $M_s = 120^\circ\text{C}$  (Cu-24.08at%Zn-8.102at%Al). Crack initiation at variant boundaries was also found if the thermal cycling through the transformation preceded the mechanical cycling. An example has been presented for an Cu-38.84at%Zn alloy ( $M_s = -7^\circ\text{C}$ ), which was mechanically cycled after 30 thermal cycles. It is not clear till now if microcracks that appear at variant boundaries are formed by the same mechanism which controls the nucleation of the habit plane defects in single crystal specimens.

b) A second way to avoid grain boundary induced fatigue cracking is a change in grain boundary morphology. After betatizing a Cu-Zn-Al polycrystal by heating once more to the betatizing temperature at which it is rolled, and by subsequently water quenching produces boundaries with rugged microstructure as shown in fig. 4. This affects the formation of dislocations and segregation in its environment in such a way that no grain boundary cracking is observed. It has been shown that both mechanisms a and b, increase specimen life by about the same amount, but less than one order of magnitude.

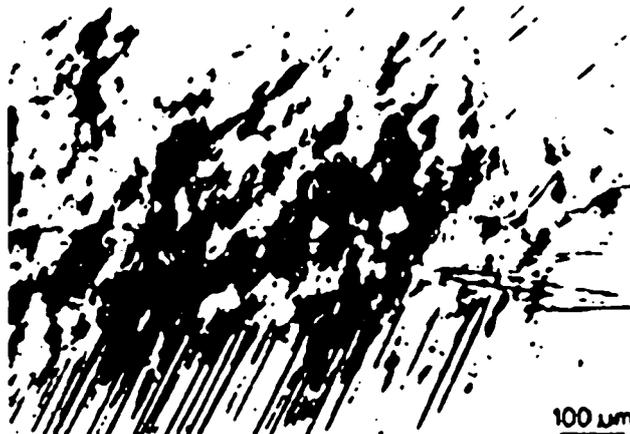


Fig.4: Highly deformed  $\beta$ -boundary. Slip steps do not interact with grain boundaries. Fracture is now intracrystalline (17)

### Discussion

The essence of the experimental results is that the behavior of single crystals is similar to the behavior of polycrystalline material if intercrystalline embrittlement is removed. This is possible if the alloys remain in the martensitic state or if the amount of deformation is sufficiently small and allows single crystal defects to nucleate before high stress

concentration in grain boundaries accumulate. Therefore, studies of single crystal are worthwhile for the fundamental understanding of fatigue mechanisms and fatigue life of shape memory alloys.

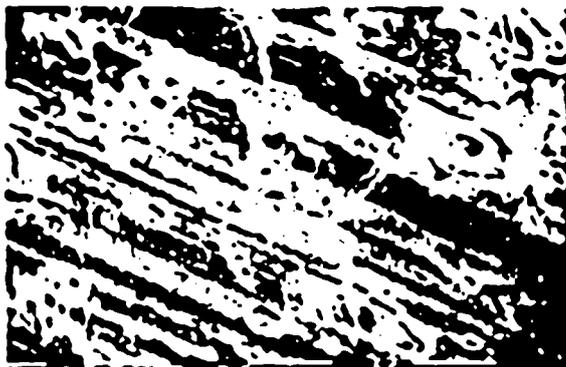
Special attention has been paid to the formation of surface defects, mainly to intrusions-extrusions during pseudoelastic cycling. These habit plane defects are considered to be the origin of microcracks both in pseudoelastic cycling under tension and under compression stress, in single crystals. Experiments performed with polycrystalline specimens have shown the same type of surface defects if the deformation is kept low enough. The influence of this microcrack formation has been mostly overlooked in experiments which were performed with polycrystalline specimens. The reason is that crack nucleation and fracture at  $\beta$ -grain boundaries has been the normal mechanism conducting to crack nucleation and fracture in Cu based alloys.

The following path can be suggested for the formation of these habit plane defects due to pseudoelastic cycling, if the dislocation structure and surface phenomena are considered:

1. Stress induced formation of a martensite crystal of width which corresponds to the length of the hole which is observed in a later stage.
2. Plastic deformation in the interface and on the slip systems in the interior of this martensite crystal. It should be mentioned here that dislocations found in the  $\beta$  phase after pseudoelastic cycling lie in the habit plane of the transformation and in the plane which transforms to the basal plane of the martensite (13).
3. During the M- $\beta$  reversion the intra-martensitic plastic deformation is pulled back. Similar to external tension-compression cycling in classical fatigue this reversion of plastic deformation is not quite complete.
4. The variant interface limits sideways growth of the slip step during subsequent cyclic tension or compression. Repeated reversion of intramartensitic plastic deformation produces the tongue-like extrusion and rectangular hole as a consequence of limited cross-slip and therefore incomplete plastic reversibility. A support for the suggested mechanism is found in fig.5.A SEM picture shows a single crystal under tensile load. The dark bands are stress induced martensite and the light ones are  $\beta$ . The intrusions-extrusions lie on the martensitic bands and they are limited by the  $\beta$ -M boundary.

It should be noted that surface treatments applied to single crystals specimens have shown that a considerable increase in fatigue life can be obtained if intrusion-extrusion type defects are eliminated at intermittent stages of cycling (3). On the other hand, grain boundary brittleness can be removed after an adequate thermomechanical treatment, leading also to an increase in fatigue life. To which extent, this and other possible treatments can be combined and applied to polycrystalline material, will depend on the type of fatigue involved. As an example removal of habit plane defects and modification of grain boundary morphology should be required in the pseudoelastic temperature range.

If thermally induced martensite is mechanically fatigued, the role of  $\beta$ -grain boundaries is smaller due to the martensite variant



**Fig. 5:** S.E.M. picture of a partially stress induced single crystalline sample. Dark bands are martensite. Intrusion-extrusion defects are present in the martensitic bands, limited by the  $\beta$ -M boundary. 6162 cycles were previously performed at  $T_{exp} = 60^{\circ}C$ . Bar indicates  $10\mu m$

boundaries, which act as new sites for cleavage. Intramartensitic deformation is also present in this case (7). Deformation mechanisms found in single crystals should play a dominant role here. Intramartensitic deformation can be related to defects lying in basal planes of martensite, which were found in single crystal due to pseudoelastic cycling (3). However these "basal plane defects" played a minor role in microcrack nucleation, which was mostly connected with habit plane defects.

### Summary

Consequences of fatigue on single and polycrystalline specimens have been analysed. Defects which appear on the surface, which lead to microcrack nucleation, have been used in this analysis. However this can be extended to the evolution of the interior of the specimens and its relation to changes in stress deformation curves. Further research is made in this direction.

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