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YIELD DROP AND SNAP ACTION IN A WARM WORKED Ni-Ti-Fe ALLOY

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The tensile behavior of a warm worked Ni-Ti-Fe alloy exhibiting both
premartensitic and martensitic transformations has been studied over the
temperature range -196 C to +40 C. Two unique mechanical phenomena were
observed: a diffuse yield drop, and "snap action". A theory is presented
to account for these observations whereby the polygonized microstructure
found after warm working impedes the growth of small martensite plates.
The suggested "penning" mechanism delays the transformation, creating
what can be considered a supersaturated condition. When the critical
stress is reached, a much lower stress is required to continue the deform-
ation. The result is an instability in strain, resulting in a snap-action
which is heating rate and strain rate independent, and is too rapid to
record with high speed x-y recorders. Penning also occurs during reversion,
thus the snap is also found during recovery and pseudoelastic unloading.

I. Introduction

Numerous investigations of the alloy Ti(50)Ni(47)Fe(3) have been conducted in
regard to both its premartensitic and martensitic transformations. The alloy is
particularly well suited for studying premartensitic effects because of the large
separation between the two transformations [1,2]. In the present work the effects
of a dislocation substructure on the nucleation and growth of the martensitic and
premartensitic phases introduced by warm working were investigated. It will be
shown that the presence of a substructure leads to a new type of yield phenomenon
and a step function memory effect called "snap action".

Material of nominal composition Ti(50)Ni(47)Fe(3) was prepared using standard
techniques for NiTi based alloys [3]. Conventional hot worked bar was given a
final swaging reduction below the red heat range to produce the dislocation cell
structure shown in Figure 1. Tensile specimens 6.35mm in diameter were machined
from this warm worked bar and given a stress relief heat treatment. All mech-
anical testing was performed on a computer controlled, servo-hydraulic test
machine in an environmental chamber capable of controlling temperatures between
-196 C and +300 C. An engineering strain rate of 0.001 /second was used unless
noted otherwise. M_S in this alloy was measured via electrical resistivity to be
below -196 C (estimate from other techniques to be -210 C), and the R-phase
transformation temperature (T_R) was found to be -40 C.

II. Results

(1) General tensile behavior of warm-worked NiTiFe

The stress-strain curve in Figure 2 is consistent with the fact that warm
worked NiTiFe exhibits both a premartensitic and martensitic transformation for
deformation at -196 C. This behavior is typical for test temperatures below R_d and
M_d (the temperatures above which the R-phase and the martensite respectively can
no longer be stress induced). The first plateau is associated with the rearrange-
ment of the R-phase variants as evidenced by the fact that the test temperature is
well below T_R [4]. The R-phase plateau extends approximately 1.5% in strain. This
is greater than that which has been reported for fully annealed Ni-Ti alloys [5].

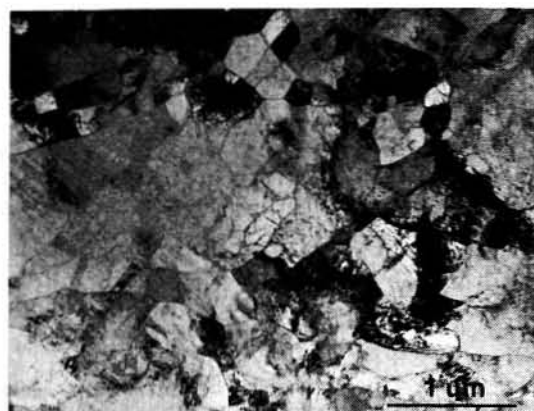Raychem Corporation, 300 Constitution Drive, Menlo Park, CA 94025   USA

Figure 1: Electron micrograph showing the fine sub-grains of the alloy studied.
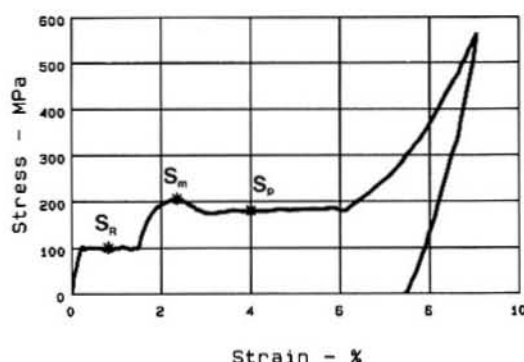
Figure 2: Strain controlled stress-strain curve conducted at -196 C.

The first plateau is followed by a stress increase and subsequent hump-like yield drop. Unlike the familiar abrupt yielding found during Lüder's deformation, this yield phenomenon is diffuse and is immediately followed by the plateau associated with stress inducing the martensitic phase. The premartensitic plateau stress S_R, the maximum stress of the yield peak S_M, and the martensitic plateau S_p were found to exhibit the temperature dependence shown in Figure 3. It was found that the stress level for the premartensitic plateau decreased with increasing temperatures from -196 C to T_R, indicating that deformation in this range is a thermally activated process. On the other hand, the stress needed to induce the martensite increases with increasing temperature in conformance with the Clausius-Clapeyron equation ($dS/dT = \Delta H/T \Delta \epsilon$) [6]. Of particular importance is the fact that the maximum stress of the yield point also increases with increasing temperature and closely follows the dS/dT behavior of the martensitic plateau. This verifies that the yield point is related to the thermo-elasticity of the martensite. If the yield point were attributable to a traditional thermally activated deformation mechanism, a temperature dependence opposite to this would be expected. The strain rate dependencies of S_R, S_M, and S_p were also investigated at -196 C. No clear relation between any of these and the rate of deformation was detected up to strain rates of 0.01 /second. Differences attributable to such a dependence if it does exist, were overshadowed by variability from one test specimen to another.
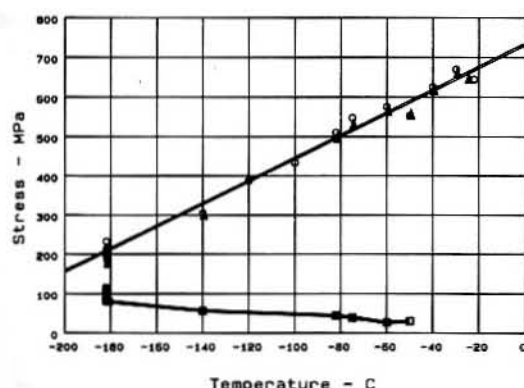

Figure 3: Temperature dependencies of R-phase strength (squares), martensite peak (circles) and martensite plateau (triangles).
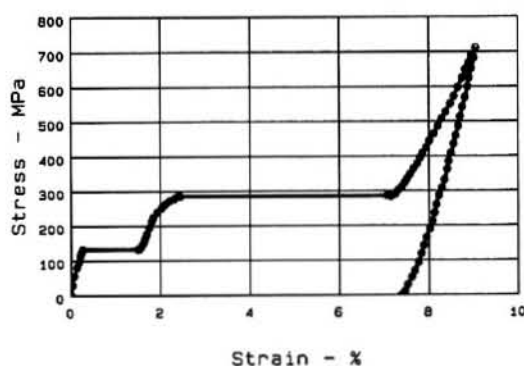
Figure 4: Stress controlled stress-strain curve showing the strain discontinuity (snap action) during deformation.

(2) Snap-action during deformation

Figure 2 was obtained by monotonically increasing strain (a so-called strain-controlled test). A unique discontinuity is observed if instead, stress is the controlled parameter. In Figure 4, stress was monotonically increased at a rate of 5 MPa/second . There is essentially no difference between Figures 2 and 4 until the yield peak preceding the martensitic plateau. At this point however, there is a discontinuity in strain; an incremental stress increase results in a very large increase in strain (3 to 4 percent). Thus the specimen "snaps" at very high velocities to a new geometry. This snap is quite audible and occurs at a speed apparently controlled by the inertia of the test system.

(3) Strain recovery after deformation

The strain associated with the deformation of both the martensite and R-phase can be fully or partially recovered by the application of heat. This "recovery" is the well known and documented shape memory effect in Ni-Ti alloys. The strain recovery profile (strain vs. temperature) for subsequent unloading and heating of the specimen shown in Figure 2 is shown in Figure 5. The two distinct regions where changes in strain occur correspond to the premartensitic (R-phase) and martensitic transformations. More significant to this study however, is the observation that the first recovery, at -124 C, occurs with the same snap action detected for the deformation process. The magnitude of this discontinuity was 5%. Recoveries were profiled at several heating rates (as slow as 1 C/min.) and the snap was found to be rate independent. The velocity of the snap was too rapid to measure using conventional data acquisition techniques and high speed x-y recorders; efforts using more rapid techniques are underway.

The above cycle of deforming at a constant temperature and heating to recover the strain is commonly used to study and demonstrate the shape memory effect. In practice, a more common method of deforming is to apply a constant stress and cool, the deformation occurring as T_R and M_S are reached. This is the case for mechanical actuators where the deformation "sets" the device and the application of heat triggers useful motion [7]. A test analogous to this situation is shown in Figure 6. Here the change in strain with temperature on thermally cycling at constant stress is shown for temperatures between 0 C and -196 C. The most interesting observation from this test was that the transformation from the R-phase to martensite on cooling under a 350 Mpa stress occurred with the same instability noted above for tensile deformation and recovery. Furthermore, the strain recovery when heating under constant stress also exhibited snap-action. Such behavior has not been reported previously. Referring again to mechanical actuators, one can visualize a device that would snap back and forth between two points with changes in temperature. Stresses in excess of 400 MPa were found to suppress the "snap" motion.
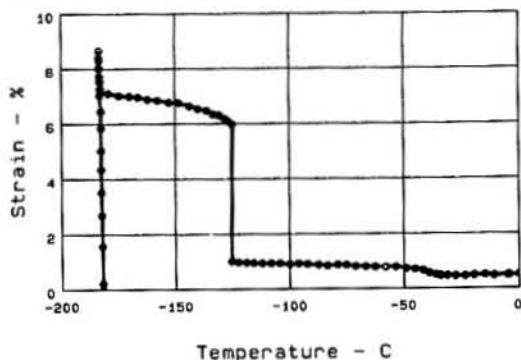

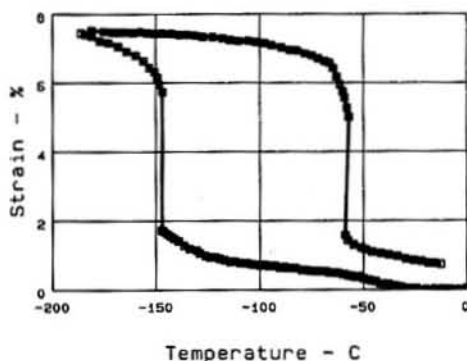Figure 5: Strain recovery profile after deforming 9% at -196 C (shown in Fig 2).

Figure 6: Strain response as the alloy is cooled and then heated while subjected to a 350 MPa tensile stress.
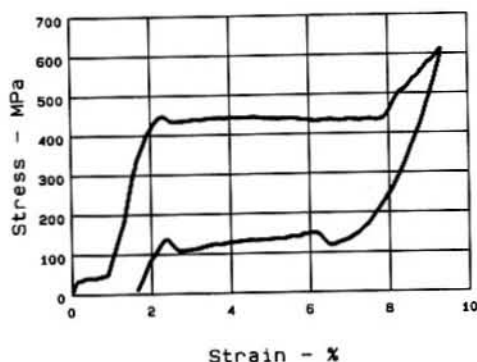
Figure 7: Tensile test conducted in strain control at -120 C (in the pseudo-elastic range for this alloy). Note the strain instabilities during both loading and unloading.

(4)   Snap-action in the psuedoelastic range

     In the tensile experiments described above, unloading occurred below A_S. Furthermore, snap action was observed throughout the temperature range from -196 C to -75 C. Unloading between A_S and M_d leads to psuedoelasticity. At temperatures within the psuedoelastic range which are also within the range where snap-action was observed, recovery of strain upon removal of the load also occurred in a "snap" fashion. The snap was again audible and its speed apparently controlled by the inertia of the test system. Unloading in these cases was conducted by monotonically decreasing the stress. If instead strain is monotonically decreased, the unloading curve in Figure 7 is obtained. Note the reappearance of a hump in this unloading curve. This means that an increase in stress is needed to further "unload" the specimen. A second hump between 2 and 3 percent strain was also consistently observed when unloading was performed in strain control.

(5) Structure at the yield point

     That martensite is formed at the onset of the diffuse yield point was verified by interrupting the deformation in the middle of the hump. The specimen was unloaded (still at -196) at approximately 2% strain as shown in Figure 8 and then allowed to freely recover. Strain and temperature were recorded as before for this recovery process, and again two distinct transformations were detected. The first occurred at -170 C for the martensite to R-phase transformation and the second at -40 C for the R-phase to austenite transformation as shown in Figure 9. The implication of this observation is that the yield drop is not a nucleation phenomenon.
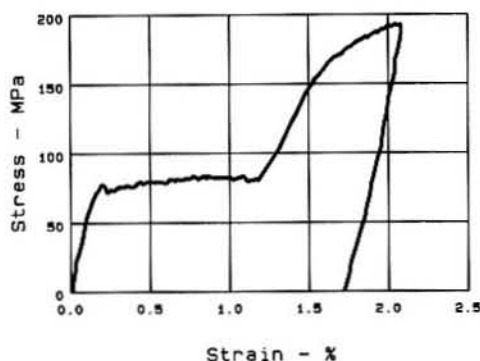

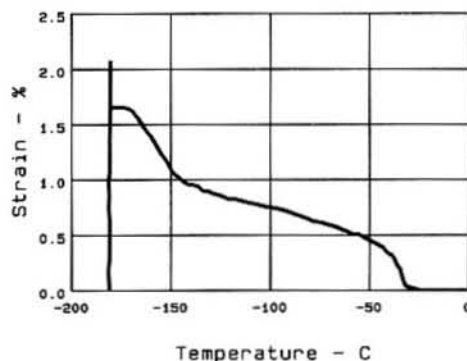Figure 8: Strain controlled stress-strain curve stopped at the yield maximum (magnified from Figure 1).

Figure 9: Strain recovery path after the deformation shown in Figure 8.

(6) Other Observations

In addition to the results reported above, several other observations were made. First, after deformation and recovery, tensile tests could be immediately repeated and the same yield phenomena found. Second, snap-action was not observed on heating if the deformation was halted before the end of the martensitic plateau. In addition, unloading at the middle of the martensitic plateau and reloading without first recovering did not result in a reappearance of the yield drop. Finally, snap-action has been observed in other ternary Ni-Ti alloys which do not show an R-phase.

III. Mechanism of the yield drop phenomenon

A successful hypothesis for the mechanism of the yield drop and snap-action must explain the following observations:
1. The yield drop is diffuse and without the serrations typical of strain ageing.
2. The phenomenon is observed in both directions, i.e. parent phase-to-martensite and martensite-to-parent phase.
3. The behavior is not found in the annealed condition.
4. To a first approximation, the yield drop is strain rate insensitive and snap-action recovery is heating rate independent.
5. The yield point follows the martensite plateau with respect to deformation temperature.
6. The magnitude of the snap is large (as high as 5%).
7. Deformation to at least the end of the martensite plateau is necessary to produce the instability during reversion.
8. Unloading and reloading at the middle of the plateau without first recovering does not cause a reappearance of the drop (as in strain ageing).

We can examine some of the "conventional" explanations of yield phenomena with respect to the above observations:
1. Pinning by deformation debris: If the deformation debris from the warm working process were to interact with the martensite plates, one might expect a serrated yield, but not a diffuse drop. We are unable to explain why pinning would allow some 0.5% or more deformation before having maximum effect.
2. Lüder's deformation: In the stress induced martensite regime, a shape memory material can only initiate deformation by forming localized bands extending through the complete cross-secton of the material. It is easy to visualize, therefore, that the nucleation event would be difficult and could require a supersaturation. Still, experience argues against a diffuse Lüder's yield. Maximum difficulty is encountered in trying to nucleate the first plates, not during the growth process. The explanation also fails to explain why the phenomenon is not found in the annealed condition.
3. Autocatalytic nucleation: If the nucleation event were particularly difficult in the warm-worked condition, one might expect an autocatalytic nucleation effect in which the nucleation of a first plate encourages nucleation of new plates in that vicinity (due to a localized increase in strain energy) [8]. Again, however, this is a nucleation problem, and fails to explain a yield drop during the growth stages, and again the phenomenon is only found in the warm-worked condition in which we expect an abundance of nucleation sites.

We suggest instead that all of the observations can be explained by a penning concept; "penning", because we envision the sub-grain boundaries to be somewhat flimsy barriers to the shear transformation, and that the sub-grains themselves are pens from which the shear transformation must break free in order to progress. Once growth extends beyond the first pens, the boundaries are ineffective in stopping the transformation; by analogy, a bull can be contained by a low fence if the pen is small, but once he is able to take a running start, the low fence is ineffective.

The onset of yielding is caused by scattered nucleation and growth events contained within the pens; since the average plate size far exceeds the pen size, relatively little material is transformed compared to the fully annealed condition. Thus stress must be increased beyond thermoelastic equilibrium to continue the transformation. Finally the supersaturation is sufficient to force plates through the low-angle boundaries. When the growing plate encounters the next boundary it is larger, more stable, and better able to penetrate. Thus less stress is required to continue growth, until finally the pens are of negligible consequence, and growth proceeds in the conventional thermoelastic fashion.

Though we offer no proof of the mechanism, there is empirical support. The diffuse nature is expected since the concept allows for some transformation prior to reaching a maximum stress, then allows for a gradual breakaway (since the plate would continuously become more stable with growth). To explain that the instability exists during reversion to the parent phase we must assume that the plates do not retreat, but instead that the parent phase must be nucleated and grown by a shear process similar to the development of martensite; in this case, one would expect penning in both directions. Related to this is the observation that the martensitic transformation must be completed in order for the reverse snap to occur. When deformation is interrupted prior to completion, the parent phase can grow without nucleation and no instability should exist.

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