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Some Experimental Results on Proportional and Non Proportional Tensile-Torsional Loading Tests on CuZnAl Shape Memory alloys and Modelling

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ABSTRACT : Some tension-torsion loading experiments on thin tube specimens of a Cu Zn Al polycrystalline shape memory alloy have been performed with a specific experimental device. Proportional loading tests permit to verify the normality rule for the pseudoelastic strain rate and allows the experimental validation of the thermodynamical model of pseudoelastic behaviour developed by Raniecki et al. Non proportional loadings show how the pseudoelastic behaviour depends on the chosen stress path. The chosen training path seems to have little effect on the obtained efficiency values which are very high (around 70-80 %). A microstructural experimental study is presently done to understand the mechanism of formation and reorientation of martensite plates when the stress vector direction changes.

Introduction

Until now, mechanical models of, for instance, pseudoelastic behaviour, used to be written and also valided only on the 1D direction of the uniaxial stress. Nevertheless, it is necessary to understand the S.M.A. behaviour under a multiaxial loading since it is the case in the most of industrial devices. Some tests on mechanical structures have been performed for "complex" loadings : thin rectangular plates loaded in torsion (1), thin rectangular plates loaded in flexion by a terminal force (2), springs loaded in "tension" (2) or in compression (3),... But these tests were realized either in order to study the efficiency of training, either in order to analyse the microstructure evolution.

In (4), B. Raniecki et al. write a three-dimensional model of the pseudoelastic behaviour of S.M.A.. In order to verify some of their hypothesis, some tridimensional (3D), or at least bidimensional (2D) loading tests have to be performed. The simplest bidimensional loading to realize is a tension-torsion one. Some similar experiments on SMA have been presented in (5) but samples were rigid bar specimens (Cu Al Zn Mn) which are associated with an important shear stress gradiant in torsion.

To have a more simple stress tensor, tension-torsion proportional loading tests on thin tubes have been carried out and presented in this contribution. It must be noticed that some results on this kind of test are presented in (6), but these experiments were performed on Ni Ti polycrystal and they still are quite qualitative. More over, there are few tests examinated.

In this paper some non proportional loading tests are also exposed, to show the importance of loading sequence. Training of samples during these tests have been quantified. Finally, the modelling described in (4), is applied on a proportional loading test.

I. Experimental process

The tests have been performed on a Cu Zn Al polycrystalline S.M.A. without any additional component (weight composition : Cu 70.17 %, Zn 25.63 %, Al 4.2 %). Its characteristic phase transformation temperatures, determined by home electric measurements are : M_S=287 K ; M_F=278 K ; A_S=290 K, A_F=293 K.The heat treatment is quite classical. Samples are heated at 1123 K during 15 minutes, quenched in a 393 K oil bath and maintained at this temperature during one hour. The samples are carried out few days later, in order to make the austenitic phase more stable.

The technical process is well described in (7). The sample dimensions lead to use the thin tube condition. The stress tensor is, with this assumption :

$$\underline{\sigma} = \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & \sigma_{z\theta} \\ 0 & \sigma_{z\theta} & \sigma_{zz} \end{pmatrix} \qquad (1)$$

with

$$\begin{cases} \sigma_{zz} = F / 2\pi \, R \, e \\ \sigma_{z\theta} = C / 2\pi \, R^2 \, e \end{cases} \qquad (2)$$

F, C, R and e are respectively the axial loading force, the torque, the mean radius and the thickness of the sample. It must be noticed that in the S.M.A. case, the thin tube condition must take into account the mean grain size which is about 1 mm. Some 1800 grains have been numbered in the useful part.

Tests are performed on a Schenk 3D test machine (tension, torsion and internal pressure), plotted by an H.P. microcomputer. Every test has been realized with a force control. Temperature is maintained constant at T = 303 K (T> A_F). Stresses are calculated from F and C values. The axial and torsional small strains ε_{zz} and $\varepsilon_{z\theta}$ can be easily deduced by :

$$\begin{cases} \varepsilon_{zz} = \Delta L / L \\ \varepsilon_{z\theta} = \dfrac{r\,\Delta\theta}{2L} \end{cases} \qquad (3)$$

ΔL and $\Delta\theta$ are the axial and the angular displacements measured by linear and rotative sensors (L.V.D.T., R.V.D.T.). L is the active length.

II. Experimental results

In first place are performed some proportional loading tests. In this case, the equivalent stress, in agreement with Von Mises criterion, is defined as :

$$\overline{\sigma} = (\sigma_{zz}^2 + 3\sigma_{z\theta}^2)^{1/2} \qquad (4)$$

For each proportional test, the maximum equivalent stress $\overline{\sigma}_{max}$ is 110 MPa. As the loading is proportional, axial and torsional stresses are linked each other ($\sigma_{z\theta} = \alpha\,\sigma_{zz}$ where α, fixed in each test, characterizes the direction of loading and can vary between 0 -pure tensile test- and ∞ -pure torsional test-). Test frequency is 10^{-3} Hz. Loading and unloading periods are similar. The first cycle is repeated 35 times in order to evaluate the possible training of the samples.

The stress-strain curve corresponding to the test with α = 0,333 is presented *figure 1*, as a representative test. In (7), the study of the phase transformation yield stress value versus α evolution leads to define a criterion surface as $\overline{\sigma}^{AM}$ = 30.3 Mpa. Generalizing the

1D relation $\sigma^{AM} = b(T - M_S)$, the constant b is found to be equal to 1.9 MPa.K^{-1} which is a classical value for a Cu Zn Al polycrystal. ... (8), uniaxial tests with the same alloy were presented and b was estimated to 2.0 MPa.K^{-1}.

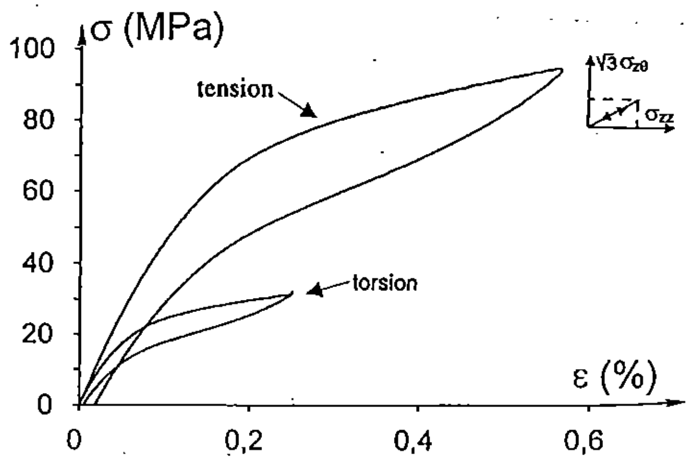


fig.1 : Tension-torsion proportional loading test (α=0.333)

And what about the pseudoelastic behaviour? For tensile tests, Vacher in (8) has established the proportionality between the pseudoelastic deformation ε^{pe} and the volume fraction of martensite z by electrical resistance measurements during mechanical tests. For a 2D or a 3D proportional loading, as in "plasticity", the existence of a current flow surface $(\overline{\sigma} = cte)$ is postulated. It is homothetic to the initial one $(\overline{\sigma}^{AM} = cte)$; the normality rule i.e. the pseudoelastic strain rate is perpendicular to this surface. In (7), an integration of the pseudoelastic strain rate gives ;

$$\underline{\varepsilon}^{pe} = \frac{3}{2} \frac{\text{dev}\,\underline{\sigma}}{\overline{\sigma}} \gamma\, z \qquad (5)$$

where γ is the maximal pseudoelastic strain obtained for a complete phase transformation occuring in a tensile test.

To verify the validity of this expression, the evolution of $\varepsilon^{pe}_{z\theta} / \varepsilon^{pe}_{zz}$ is proposed. In that way, a parameter Q is defined as :

$$Q=\frac{\varepsilon_{zz}^{pe}}{\varepsilon_{z\theta}^{pe}}\cdot\frac{\sigma_{z\theta}}{\sigma_{zz}} \qquad (6)$$

From (5), Q is theoretically constant and equal to 2/3. Q mean values for three biaxial loading tests are presented in the *table 1*. It can be noticed that even Q seems to be a constant, its value is slighty higher than the theoritical one.

α	0.333	0.577	1
Q	0.72	0.75	0.75

Tab. 1 : Q value obtained for each proportional test

The equivalent pseudoelastic strain is defined by :

$$\bar{\varepsilon}^{pe}=\left(\varepsilon_{zz}^{pe2}+\frac{4}{3}\varepsilon_{z\theta}^{pe2}\right)^{1/2} \qquad (7)$$

Its maximum value is reached when $\bar{\sigma}$ is maximal $(\bar{\sigma}_{max}=110\,\text{MPa})$.The *table 2* gives $\bar{\varepsilon}_{max}^{pe}$ for each test. In a pure tension test, $\varepsilon_{z\theta}^{pe}$ is theoretically null and in a pure torsional test, ε_{zz}^{pe} also. From the *table 2*, the material seems to be slightly anisotropic. This can explain why Q is not equal to its theoretical value.

Test	0	2	0.333	0.577	1
ε_{zz}^{PE} (%)	0.339	0,011	0,384	0.369	0.193
$\varepsilon_{z\theta}^{PE}$ (%)	0,025	0,215	0.173	0.277	0.356
$\bar{\varepsilon}^{PE}$ (%)	0.34	0.248	0.433	0.488	0.454

Tab. 2 : Experimental tensile and torsional pseudoelastic strain values

The pseudoelastic strain measurements allow also to know the pseudoelastic strain rate vector $\vec{\dot{\varepsilon}}^{pe}$. As the loading is proportional, the following relation holds (from eq. (5)) :

$$\frac{\varepsilon_{z\theta}^{pe}}{\varepsilon_{zz}^{pe}} = \frac{\dot{\varepsilon}_{z\theta}^{pe}}{\dot{\varepsilon}_{zz}^{pe}} \qquad (8)$$

Then, it's easy to draw $\vec{\dot{\varepsilon}}^{pe}$ on the $(\sigma_{zz}, \sqrt{3}\,\sigma_{z\theta})$ plane, for each test, when the equivalent stress is maximal, as it is done on *figure 2.* This figure shows that the strain rate vector is quite perpendicular to the loading surface $(\bar{\sigma}_{max} = 110\ \text{MPa})$ which proves the validity of the normality rule. Moreover, it allows to see the expansion of the criterion surface from $\bar{\sigma}^{AM} = 30.3\ \text{MPa}$ to $\bar{\sigma}_{max} = 110\ \text{MPa}$.

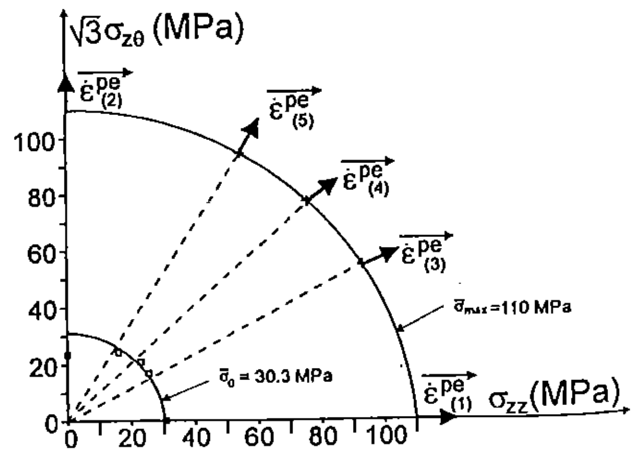
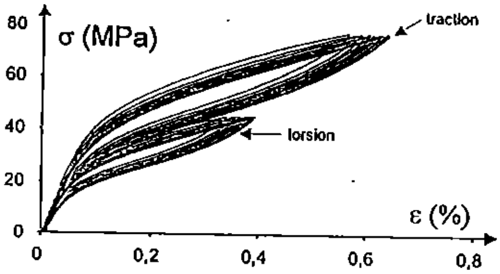


fig. 2 : Experimental validation of the normality rule

After $N_{max} = 35$ loading-unloading cycles (between $\bar{\sigma}=0$ and $\bar{\sigma} = \bar{\sigma}_{max} = 110\ \text{MPa}$), the training effect is also measured. The *figure 3* represents the ten first cycles corresponding to the test with α=0.577. The *figure 4* shows the training effect on this sample, placed (at a free stress state) in an oil bath which temperature varies from 232 K to 313 K.



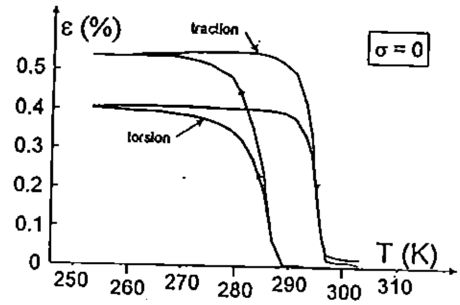


fig. 3 : the fist ten cycles of a training process (a) and the two way memory effect obtained (b)

In order to study the training effect, it is necessary to define three training efficiencies : the tension efficiency, the torsion efficiency and the equivalent efficiency (respectively $\rho_{zz} = (\Delta\varepsilon_{zz})_{\sigma=0} / (\varepsilon^{pe}_{zz\,max})_{N=N_{max}}$, $\rho_{z\theta} = (\Delta\varepsilon_{z\theta})_{\sigma=0} / (\varepsilon^{pe}_{z\theta\,max})_{N=N_{max}}$ and $\rho = (\Delta\bar{\varepsilon})_{\sigma=0} / (\bar{\varepsilon}^{pe}_{max})_{N=N_{max}}$). These efficiencies are measured for the five training tests. These values seem much higher (around 75-80 %) than the ones obtained with a more complex loading (for example in (1)) In (5), rigid bars are also trained in tension and torsion in order to study the stabilization of stress induced martensite. Unfortunately, efficiency values are not presented. Nevertheless, the pseudoelastic loop is stabilized after few cycles, as in our experiments. Training values lead to assume that the density of dislocations is quite important since in (9), it is said that the density of dislocations is a good parameter to evaluate the training effect. Actually, some microscopical analysis on biaxial tensile loading tests are being performed to wellknow the microstructural phenomena. They will perhaps verify this asumption.

In second place, it was interesting to study the effect of a non proportional loading. Even if the modelling of such tests is, in most of cases, not simple, their interest appears in answering to the question : "what is the effect of a rough change of the mechanical loading upon the material's behaviour ?" Indeed, in each single crystal, the best oriented habit planes are activated (with respect to the maximum shear rule) for a given loading direction (10). If the direction of the stress vector is changed by applying torsion above tension, other variants ("secondary variants") can be activated and interact with the primary variants. Here, the interest is to see what happen macroscopically with a polycrystalline sample.

As in proportional loading tests, the maximum equivalent stress is 110 MPa but it is reached from one of the four different possible pathes. β is an angle characterizing the test $\left(tg\,\beta = \frac{\sqrt{3}\,\sigma_{z\theta}^{max}}{\sigma_{zz}^{max}} \right)$. Its possible values are 30°, 45°, 60°. For each path, the tensile $(\sigma_{zz}, \varepsilon_{zz})$ and torsional curves $(\sigma_{z\theta}, \varepsilon_{z\theta})$ are given *(figures 4 to 7)*. So, the resulting deformation path $(\varepsilon_{z\theta}, \varepsilon_{zz})$ is known. As it was already said in the previous part, material isotropy is not perfect since during the first loading (uniaxial one), a small strain is measured along the other axis. Maximum equivalent pseudoelastic strain $\bar{\varepsilon}_{max}^{pe}$ is higher than in proportional loading tests. This observation confirms the assumption that new habit planes ("secondary" planes) are activated when the mechanical loading direction changes. Moreover, it seems that the hardening induced by interactions between primary and secondary habit planes does not play an important role. Moreover, during the second loading (BC) it can exist a reorientation of primary variants with the change of the stress vector orientation. Such variant reorientations are underlined in (5). In fact, during these non proportional loading tests, an observation of the microstructure evolution is necessary to understand the micromechanisms involved by the stress path. At the phenomenological point of view, the correspondance between the shape of the imposed stress path and the resulting shape in the deformation path is interesting.

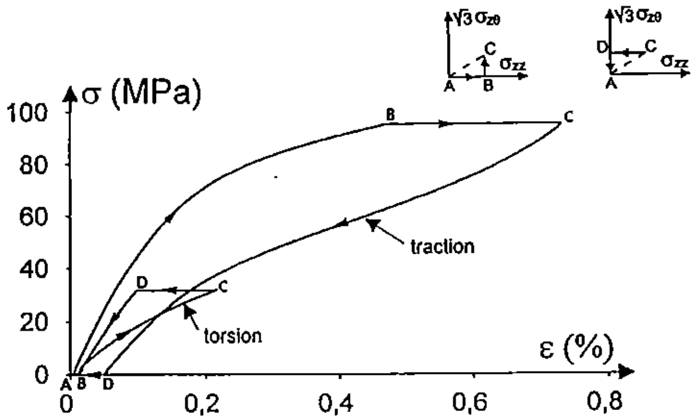


fig. 4 : Tension-torsion non proportional loading test (path I, β=30°)

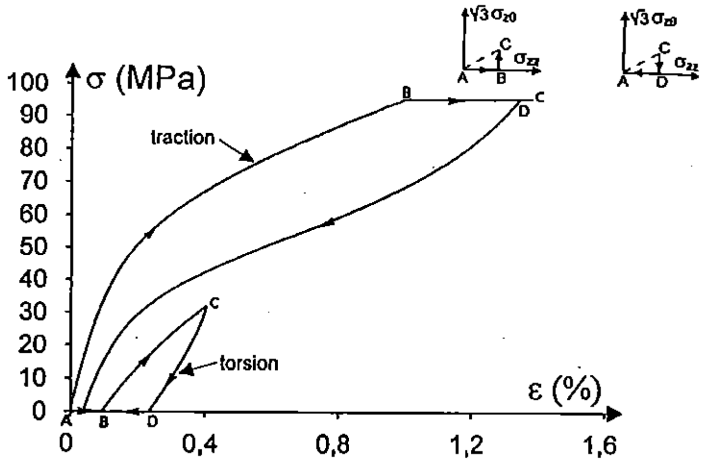


fig. 5 : Tension-torsion non proportional loading test (path II, β=30°)

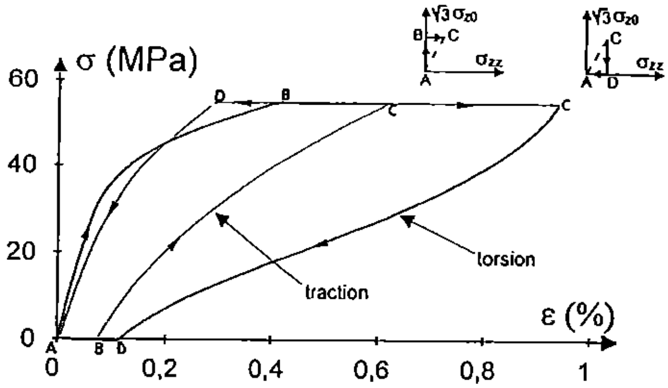


fig. 6 : Tension-torsion non proportional loading test (path III, β=60°)

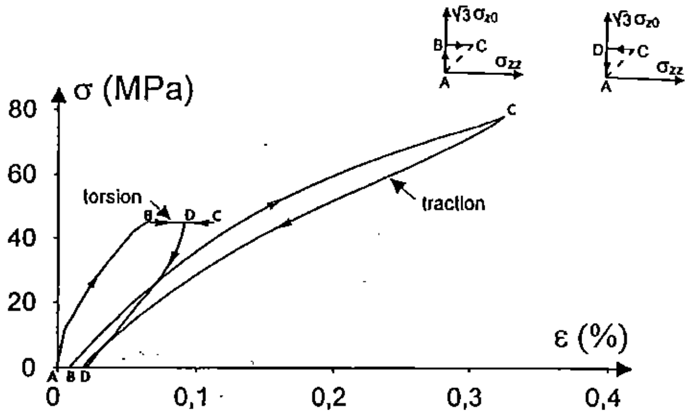


fig. 7 : Tension-torsion non proportional loading test (path IV, β=45°)

The training effect is also studied (11). The global efficiency ρ lies between 60 and 80 % but the dependance with the chosen path is not clear.

III. Modelling of a proportionnal loading test

In (4), Raniecki et al. propose to model the pseudoelastic behaviour in two steps. First the free energy of the two phases system (A+M) is written as

$$\Phi = (1 - z)\Phi_{(1)} + z\Phi_{(2)} + \Delta\Phi \qquad (9)$$

Φ_1 and Φ_2 are the specific free energies of respectively the austenite and of the martensite phases. $\Delta\Phi$ is called the configurational energy and represents the interaction which appears between the two phases, for example producted by incompatibilities between deformations. The main characteristic of this energy is to disappear if only one phases is present in the material. In agreement with Muller and Xu (12), the simplest expression is :

$$\Delta\Phi = z(1 - z)\Phi_{it} \qquad (10)$$

(Φ_{it} is the interaction energy ($\Phi_{it}(T) = \overline{u_0} - T\overline{s_0}$).

In (4), the free energy expression is obtained as (z is the volume fraction of martensite and ρ the volume masse of the material) :

$$\Phi(\underline{\varepsilon}, T, z) = u_0^1 - Ts_0^1 - z\pi_0^f(T) + \frac{1}{2\rho}(\underline{\varepsilon} - \underline{\varepsilon}^{pe})\underline{\underline{L}}(\underline{\varepsilon} - \underline{\varepsilon}^{pe}) + c_v\left[(T - T_0) - T\ln\left(\frac{T}{T_0}\right)\right] + \Delta\Phi \qquad (11)$$

with :

$$\underline{\sigma} = \rho\frac{\partial\Phi}{\partial\underline{\varepsilon}} = \underline{\underline{L}}(\underline{\varepsilon} - \underline{\varepsilon}^{pe}) = \underline{\underline{L}}\underline{\varepsilon}^e \qquad (12)$$

$$s = -\frac{\partial \Phi}{\partial T} \quad (13)$$

$$\pi_0^f(T) = (u_0^1 - u_0^2) - T(s_0^1 - s_0^2) = \Delta u - T\Delta s \quad (14)$$

π_0^f is the working force of the martensitic transformation without any stress. u_0^α and s_0^α are the specific energy and entropy of the α phase (α=1 for the austenite and α=2 for the martensite).

The thermodynamical force associated to the phase transition is :

$$\pi^f = -\frac{\partial \Phi}{\partial z} = \pi_0^f(T) + \gamma\bar{\sigma}/\rho - \Phi_{it}(1-2z) \quad (15)$$

The Clausius Duhem inequality ($\pi^f dz \geq 0$) is chosen to be the criterion of phase transition (4) :

$$\begin{cases} \text{direct transformation} & dz > 0 \quad \pi^f \geq 0 \\ \text{inverse transformation} & dz < 0 \quad \pi^f \leq 0 \end{cases} \quad (16)$$

$\pi^f = 0$ represents the absolute equilibrium states of the system. It is instable if $\Phi_{it} > 0$, which is the general case. It is then possible to determine the equivalent stress threshold of the martensitic transformation $\bar{\sigma}^{AM}$ (point A of the *figure 9*) and for the reverse transformation $\bar{\sigma}^{MA}$ (point A_1) as :

$$\begin{cases} \pi^f(\bar{\sigma}^{AM}, z=0, T) = 0 \quad \Rightarrow \quad \bar{\sigma}^{AM}(T) = \bar{\sigma}^{eq}(z=0) = \rho\dfrac{\Phi_{it}(T) - \pi_0^f(T)}{\gamma} \\ \pi^f(\bar{\sigma}^{MA}, z=1, T) = 0 \quad \Rightarrow \quad \bar{\sigma}^{MA}(T) = \bar{\sigma}^{eq}(z=1) = \bar{\sigma}^{AM}(T) - 2\rho\dfrac{\Phi_{it}(T)}{\gamma} \end{cases} \quad (17)$$

The instability of the equilibrium induces that there exist no thermodynamical relation which could give the equations of the hysteresis loop. Taking a similar framework as in the plasticity approach, the functions Ψ_1 (for the direct transformation) and Ψ_2 for the reverse one are assumed to be constant during the phase transition. $\Psi_1=0$ and $\Psi_2=0$ are the functions which represent the complete martensitic and reverse transformations (called also the « external loop »).

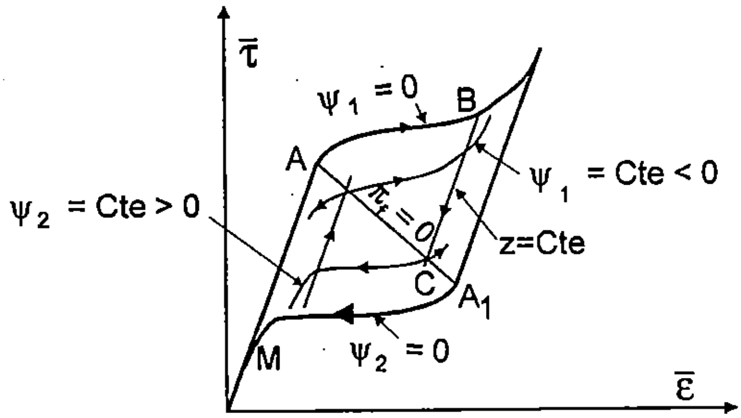


fig. 8 : Description of the external and the internal loops in the model of Raniecki and al. (4)

Ψ_1=n and Ψ_2=m (m and n are constants) represents internal loops where the transformation is not complete

$$\begin{cases} \Psi_1(\underline{\sigma},T,z)=\pi^f(\underline{\sigma},T,z)-k_1(z) \\ \Psi_2(\underline{\sigma},T,z)=-\pi^f(\underline{\sigma},T,z)+k_2(z) \end{cases} \qquad (18)$$

The functions k_1 (z) and k_2 (z) are chosen (4) such as the kinetics of the phase transformation were in agreement with the classical ones of metallurgists :

$$\begin{cases} k_1(z)=-(A_1+B_1z)\ln(1-z)+C_1z \\ k_2(z)=(A_2-B_2(1-z))\ln z-C_2(1-z) \end{cases} \qquad (19)$$

with

$$\begin{cases} C_1 = 2\Phi_{it}(M_s) & , \quad C_2 = 2\Phi_{it}(A_s) \\ a_1 A_1 = \Delta s + \overline{s_0} & , \quad a_2 A_2 = \Delta s - \overline{s_0} \\ a_1 B_1 = a_2 B_2 = 2\overline{s_0} \end{cases} \tag{20}$$

Here is now presented an application to a tension-torsion proportional loading test. The behaviour is elastic as long as the equivalent stress has not reached the critical equivalent stress $\overline{\sigma}^{AM}$ (z=0) :

$$\overline{\sigma}^{AM} = (\sigma_{zz})^{AM}\sqrt{1+3\alpha^2}\ . \tag{21}$$

Then the pseudoelastic behaviour must be simulated. The volume fraction of martensite is increasing from 0 to z_d, which is the z value obtained just before the unloading. The pseudoelastic flow is represented by Ψ_1=0. It gives :

$$\pi_0^f + \gamma\overline{\sigma}/\rho - \Phi_{it}(1-2z) = k_1(z) \tag{22}$$

As during the whole test, tension and torsion stress are proportional ($\overline{\sigma} = (\sigma_{zz})\sqrt{1+3\alpha^2}$ with $\sigma_{z\theta} = \alpha\sigma_{zz}$), it's possible to know the stress values by :

$$\begin{cases} \sigma_{zz} = \dfrac{\rho}{\gamma(1+3\alpha^2)}\left[k_1(z) + \Phi_{it}(1-2z) - \pi_0^f\right] \\ \sigma_{z\theta} = \alpha\sigma_{zz} \end{cases} \tag{23}$$

The corresponding strains are splitted into two parts as :

$$\begin{cases} \varepsilon_{zz} = \dfrac{\sigma_{zz}}{E} + \gamma z \\ \varepsilon_{z\theta} = \sigma_{z\theta}(\dfrac{1+\upsilon}{E} + \dfrac{3\gamma z}{2\overline{\sigma}}) \end{cases} \tag{24}$$

The reverse transformation is represented by the $\Psi'_2=k_2(z_d)$ curve ($k_2(z_d)$ is a negative constant) where z_d is the volumic part of martensite at the end of the loading. So, during the unloading and until the stress σ_1, the stresses are given by :

$$\begin{cases} \sigma_{zz} = \dfrac{\rho}{\gamma(1+3\alpha^2)}\left[k_2(z) - k_2(z_d) + \Phi_{it}(1-2z) - \pi_0^f\right] \\ \sigma_{z\theta} = \alpha\sigma_{zz} \end{cases} \quad (25)$$

Strains expression are still given by the equation (24).

The seven parameters A_1, B_1, C_1, A_2, B_2, C_2 and γ which determine the k_1 and the k_2 functions, Φ_{it} and π_0^f are deduced from tension loading tests described in (2) by the following constants.

Δu ($J.kg^{-1}$)	Δs ($J.kg^{-1}.K^{-1}$)	$\bar{u}_0$ ($J.kg^{-1}$)	$\bar{s}_0$ ($J.kg^{-1}.K^{-1}$)	γ	a_1 (K^{-1})	a_2 (K^{-1})
6 944	23.36	1 495	4.22	0.0416	0.032	0.06

The modelling of the test (α=0.577) is presented in the *figure 9*. The form of the loops are growthly acceptable but some ameliorations are necessary. Nevertheless, it permits to valid the 3D model proposed in Raniecki and al. in (4) and overall the state equation of pseudoelasticity (5).

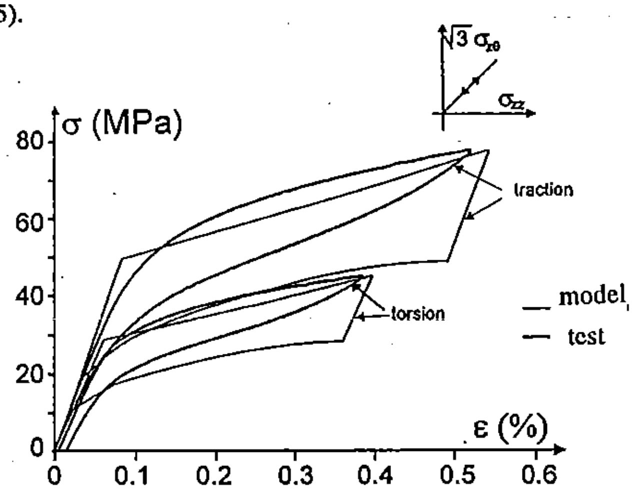


fig. 9 : Experimental and modelled curve of a proportional test (α=0.577)

Conclusion

From the proportional and non proportional tests presented here, the number of experimental data in complex loading case has increased. Proportional loading tests permit to verify the normality rule for pseudoelastic strain rate and hence allows the experimental validation of the thermodynamical model of pseudoelastic behavior developed by Raniecki et al. in (4). Non proportional loading tests brought a lot of informations not easy to interpretate. It shows the evidence that the pseudoelastic non linear behavior depends on the chosen stress path. The main physical features are the creation of new variants (called "secondary" ones) when the stress direction change, or (and) the reorientation of the first variants under the stress. For an isothermal pseudoelastic cycling (N_{max} = 35), both proportional and non proportional training processes present very high efficiency values : this is a very good information for technical applications.

Some biaxial tensile loading tests are actually performed. The hope is in observing the creation and, perhaps, the reorientation of martensite plate. These results will be very usefull for explain the present study.

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