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# BEHAVIOUR OF A 17-12 SPH STAINLESS STEEL UNDER CYCLIC, UNI AND BI-DIRECTIONAL ANISOTHERMAL LOADINGS

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## ABSTRACT

It is shown in this article that a knowledge of the mechanical behavior of the stainless steel 17-12 SPH for different isotherms ($20 \leq T \leq 650°C$) is insufficient to correctly describe its behaviour under anisothermal thermomechanical loadings. Indeed, this alloy possesses a temperature history memorization effect at intermediate temperatures.

This phenomena is studied in detail with experiments performed under uniaxial and biaxial mechanical loadings (in phase or out of phase tensile-torsion tests) for different isotherms belonging to judiciously chosen temperature history sequences. The results of cyclic torsion tests under anisothermal thermomechanical conditions are presented where strain and temperature loading evolve simultaneously in phase, in phase opposition, in 1/4 and 3/4 phase. The analysis of the totality of results shows that the material memorizes the maximal value of the cyclic stress peak reached in the zone where the derivative of the maximal cyclic stress with respect to the temperature is positive. This observation can also be applied to both uniaxial or biaxial cyclic tests. From a physical point of view, this behavior is closely related to the interaction phenomena between dislocations and point defects in insertion solid solutions.

A bibliographical study of different materials tested under anisothermal conditions confirms this observation.

## 1 - INTRODUCTION

The industrial materials used for the construction of certain components of modern thermal machines are often submitted to severe thermomechanical loadings and the precise knowledge of the behavior and the development of the isothermal and anisothermal laws proves to be indispensable for an eventual prediction of the lifetime of these structures. Toward this end and with the considerable development of internal variable models which lead to a precise modelization of the mechanical behaviour under isothermal conditions, many recent studies attempt to enlarge their domain of application to the case of anisothermals loading [1-22].

These works demonstrate the real predictive possibilities of this type of strategy given a correct mathematical formulation for the transition of the isothermal case to the anisothermal case, namely the appearance of terms in the temperature rate $\dot{T}$ affecting the kinetics equations of different internal variables (scalar or tensorial) and resulting from the temperature dependence of the parameters in these equations [2-3] [6-8] [10] [12] [14-23].

Meanwhile, certain materials possess temperature history effects meaning that a biunivocal relation between the mechanical response and the temperature does not exist [1] [6] [9] [11-12]. In this context, in addition to the correct expression of the kinetics equations, it is necessary to understand and to analyse these history effects in order to model them in view of their integration them into the general model framework.

In the present case an austenitic stainless steel is studied between 20 and 650°C. This steel is mainly used in nuclear industry, namely in the construction of heat exchangers for fast breeder reactor. An adequate knowledge of its behaviour under isothermal conditions, including the domain of utilization (20-550°C), constitutes an essential preliminary step before the study of anisothermal loadings. The results of this analysis are found in the references [24] [25]. This article presents the second step which lies in the precise study of the response of this steel to anisothermal loadings in order to demonstrate an eventual temperature history effect.

## 2 - EXPERIMENTAL TECHNIQUES

The test specimens are obtained from slices removed from 30 mm thick sheets and hyper-quenched from 1,200°C. The microstructure is entirely austenitic and the average grain diameter is 45 µm. The weight composition of this low carbon steel manufactured by Creusot-Loire is given in table I :

Table I : Weight composition of the steel

C $\leq$ 0,03 | S $\leq$ 0,001 | P $\leq$ 0,021 | Si 0,44 | Mn 1,084 | Ni 12,3 | Cr 17,54 | Mo 2,47 | Ni 0,075 | B 0,001 | Co 0,15 | Cu 0,175 | Ti < 0,005 | Nb 0,015 | Al 0,100 | Fe bal.

Several machines have been used to perform the different experimental sequences, namely a screw tensile machine (Instron) controlled by the strain and piloted by micro-computer for the stepwise isothermal tests ; an electrodynamic torsion machine which allows the strain to be a function of the temperature signal for the real anisothermal tests ; and finally a hydraulic tensile-torsion machine (Schenk) used for biaxial tests (in and out of phase) at isothermal levels.

## 3 - EXPERIMENTAL RESULTS AND ANALYSIS

The best way to approach and analyse an eventual temperature history effect is by performing thermal cycles at different isothermal levels and successively increasing and then decreasing, or vice-versa, while having the possibility of changing the values of the cycle limits. This approach is realized for monotonic, uniaxial and biaxial cyclic loadings (in and out of phase).

### 3.1 The monotonic tests

Figure 1 shows an example of a monotonic test with successively increasing then decreasing isothermal steps. If the monotonic curves obtained for each isotherm are superimposed, it is shown that within experimental error due to the presence of the yield point return phenomena, the strain levels are identical. This means that there is no noticeable temperature history effect, an observation which is in agreement with the results of Niitsu, Ikegami [5] obtained for a 304 stainless steel.

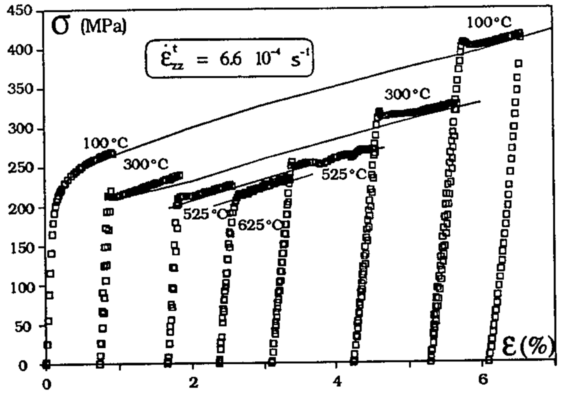

Fig. 1 : Stepwise increasing then decreasing isothermal monotonic tests.

### 3.2 Uniaxial cyclic tests

The peak values of the stress are shown in Fig. 2 for a cyclic strain test and for three cycles of increasing and decreasing temperature steps (20 ↔ 200 ↔ 400 ↔ 560 ↔ 600°C). The test characteristics are $\dot{\varepsilon}^T_{zz} = 6.6\ 10^{-4}\ s^{-1}$, $\Delta\varepsilon^T_{zz}/2 = \pm 4\ 10^{-3}$, 50 mechanical cycles for each step and a zero force control at temperatures of approximatively one hour at each step. If the average of the peak stress values is plotted for the three thermal cycles, $\Delta\sigma/2 = (\sigma_{max}$ tensile - $\sigma_{min}$ compression$)/2$, at the end of each step as a function of the temperature T, it is shown in Fig. 3 that the relation $\Delta\sigma/2 = f(T)$ is not biunivocal which means that there is a temperature history effect.

Indeed, whereas the stress levels correspond to those obtained under isothermal conditions for the first half cycle, this is not true for the remaining thermal cycles, the stress levels being larger than those of the isothermal tests. For 20°C, a hardening on the order of 80 MPa is obtained. In addition, it can be remarked that, on the one hand, the stress levels are nearly identical for the thermal cycles two and three, which means that the temperature history has been established from the first thermal half cycle, and on the other hand, the trajectories for the increasing temperature are slightly different then that of decreasing temperatures. This last point can eventually be explained by the fact that 50 cycles are not enough to obtain the stabilized level, especially in the neighbourhood of 450°C where the hardening is a maximum and also because the recentering of the cycle after the holding at zero stress is not always complete.

This experiment tends to show that the material memorizes the maximal temperature reached during the thermal cycling ($T_{max} = 600°C$) and that the hardening amplitude is a function of $T_{max}$ and of the amplitude of the domain where $n^* = (\partial(\Delta\sigma_M/2)/\partial T) > 0$. These two points are confirmed by the experiments reported in Fig. 4 and 5. Indeed, if two thermal cycles are performed starting from $T_{max} = 600°C$, it is shown in Fig. 4 that a trajectory higher than the previous experiment is obtained directly, corresponding to the material in its memorization state for $T_{max} = 600°C$.

In the experiment shown in Fig. 5, six thermal cycles are performed between 100°C and $T_{max}$ by progressively increasing the upper limit ($T_{max}$ = 200, 200, 400, 525 and 600°C). The evolution of the hardening with respect to the memorization state of the material is analysed during the transition through 100°C. For $T_{max}$ = 200°C and belonging to the zone $n^* < 0$, the cycle is reversible and no supplementary hardening appears ; for $T_{max}$ = 300°C and corresponding approximatively to $n^* = 0$, only a very slight hardening is noticeable wheareas for $T_{max}$ = 400, 525 and 600°C corresponding to $n^* > 0$, a significant hardening is obtained as an increasing function of $T_{max}$. The material thus memorizes $T_{max}$ when $n^* = (\partial(\Delta\sigma_m/2)/\partial T) > 0$.

The hardening amplitude can be evaluated by plotting a trajectory parallel to the branch $n^* < 0$, starting from the maximal temperature reached and obtained from isothermal tests. It can be shown that if the same type of experiment is performed starting from 500°C and with decreasing $T_{max}$ ($T_{max}$ = 500, 400, 300°C), then the hardening at 100°C is obtained immediately after the first stage at this temperature and its amplitude corresponds closely to cycle four of the previous experiment.

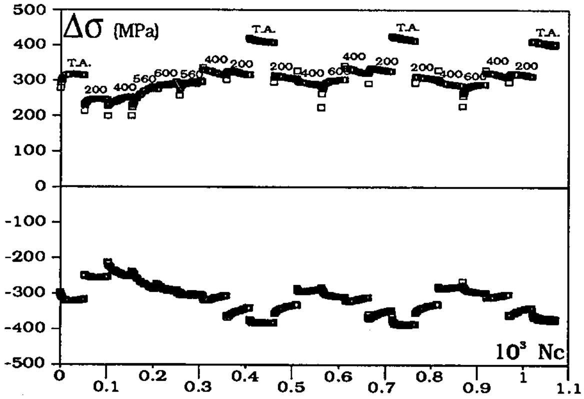

Fig. 2 : Stepwise increasing then decreasing isothermal cyclic tests. Evolution of the peak stress values.

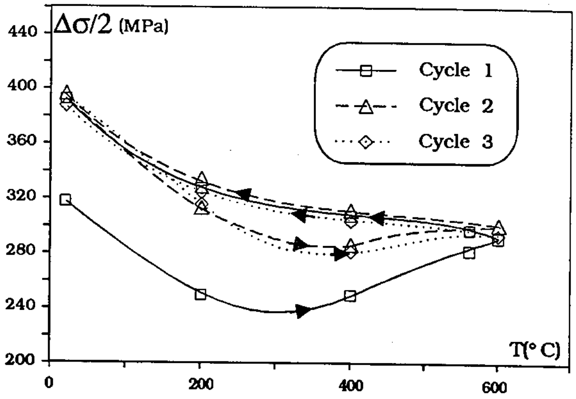

Fig. 3 : Stepwise isothermal cyclic test. Evolution of the average peak values as a function of the temperature. Demonstration of the temperature history effect.

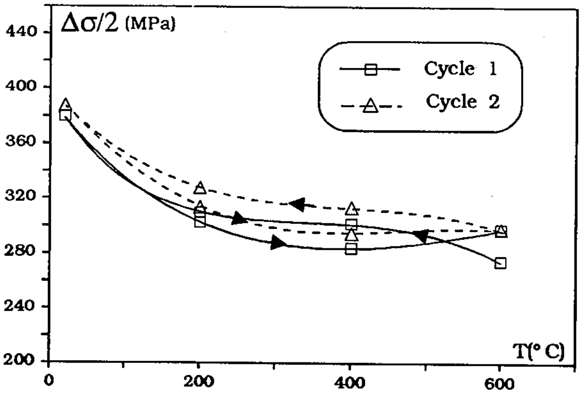

Fig. 4 : Idem Fig. 3, but starting from the maximal temperature, T = 600°C.

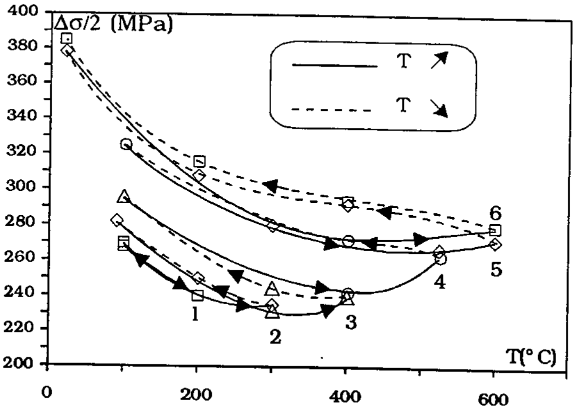

Fig. 5 : Idem Fig. 3, demonstration of the memorization of $T_{max}$ when $\partial(\Delta\sigma/2)/\partial T > 0$.

There again the material has memorized $T_{max} = 500°C$. It can also be shown that past 550-600°C, n* is negative again and that in this zone the material no longer memorizes $T_{max}$, the highest memorization temperature situated in the neighborhood of 600°C, which corresponds to n* = 0 and $(\Delta\sigma_m/2)$ maximum.

In conclusion, it can be affirmed that the memorization of $T_{max}$ is made in the domain found between the two zeros of the curve $\Delta\sigma_m/2 = f(T)$ and when its derivative is positive. Only by maintaining a high temperature ($T \geq 650°C$ for a few hours) can the hardening resulting from the temperature history be progressively erased by the recovery effect of the structure.

These different observations agree globally with those reported by Murakami et al. [11] and Ohno et al. [12], respectively on 316 and 304 stainless steel.

### 3.3 Cyclic biaxial tests in tensile-torsion

The effect of multiaxial stresses on the phenomena of temperature history memorization is studied using cyclic tensile-torsion tests, both in phase $\phi = 0°$ and out of phase, $\phi = 90°$, along different stepwise isothermal loading trajectories. It is well

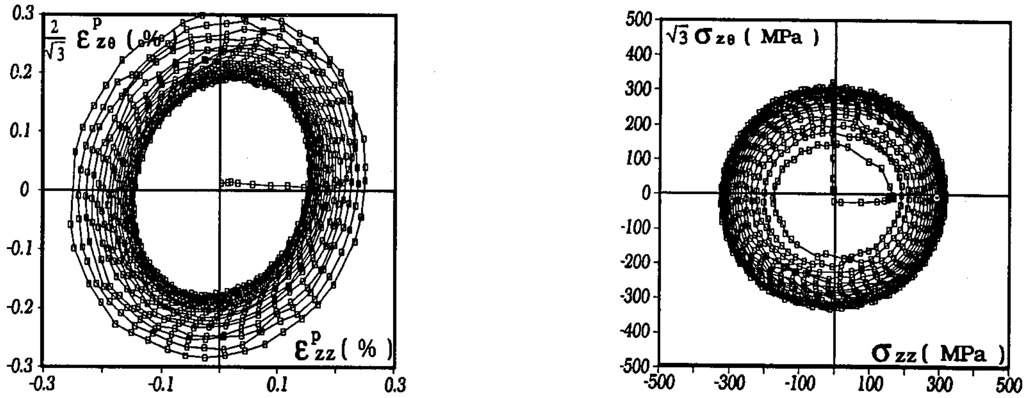

Fig. 6 : Example of cyclic hardening at 600°C for an out-of-phase ($\phi = 90°$) tensile-torsion loading.

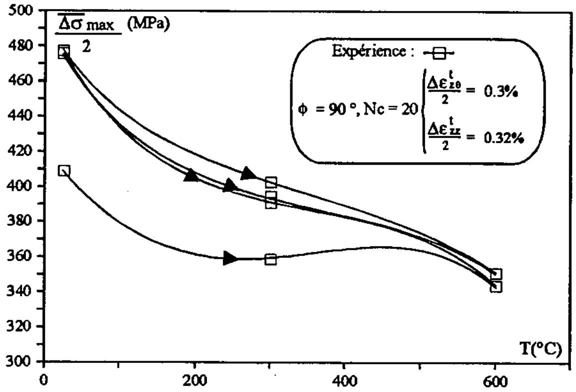

Fig. 7 : Stepwise increasing then decreasing isothermal tests with cyclic biaxial out-of-phase loadings. Demonstration of temperature history effect.

known that this steel possesses a supplementary cyclic hardening which is a function of the non-radiality of the loading and a maximum for a phase shift of 90° and a ratio between the maximal strain amplitudes equal to $R = (\Delta\varepsilon^T_{z\theta}/\Delta\varepsilon^T_{zz}) = 2/\sqrt{3}$ [26-27]. However for radial loadings ($\phi = 0°$), no supplementary hardening appears.

Since the objective is to analyse the consequences of the temperature history effect for non-radial loadings, the same stepwise isothermal loadings are adopted as those shown in Fig. 3 and 4, with $\phi = 0°$ and $\phi = 90°$. The experimental conditions are such that :

$\varepsilon^T_{zz} = \varepsilon^T_{zz} \sin \omega t$, $\varepsilon^T_{z\theta} = \varepsilon^T_{z\theta m} \sin(\omega t + \phi)$, with $\varepsilon^T_{zzm} = 3{,}2\ 10^{-3}$, $\varepsilon^T_{z\theta m} = 3.10^{-3}$,
$\omega = 6{,}3\ 10^{-2}$, $\phi = 0°$ and 90°, each step having twenty mechanical cycles.

The results mentioned in Fig. 3 and 4 are found again, both qualitatively and quantitatively, for radial loadings, indicating that the same conclusions as before can be drawn. However, in the case of out of phase loadings ($\phi = 90°$), the stress levels

$\Delta\overline{\sigma}_{max}/2$ (equivalent Von-Mises stress) are higher than those for the radial tests, as seen in Fig. 6 for the example of a hardening at 600°C. This result is in conformity with the cyclic properties of this steel of cyclic out of phase loadings under isothermal conditions. The supplementary hardening is a decreasing function of the temperature [13] [27]. Hence, during the first thermal half-cycle in Fig. 7, the levels of $\Delta\overline{\sigma}_{max}/2$ are in conformity with those of the isothermal tests while for the rest of the temperature sequence, as for the uniaxial tests, the material memorizes its passage through 600°C (Fig. 7). At room temperature, this new supplementary hardening is on the order of 70 MPa, a value which is identical to that obtained for uniaxial loadings. It is also shown that the same type of behaviour is obtained as that reported in Fig. 4 when the temperature is decreased from its maximum of 600°C with $\phi = 90°$. It should be noted that these observations are in disagreement with those of Murakami et al. [11], who reports only a very small temperature history effect for $\phi = 90°$. In summary, it has been demonstrated that the conclusions resulting from uniaxial experiments and relating to the temperature history effects remain applicable to radial and non-radial multiaxial loadings. Two types of supplementary hardening have been clearly described at 20°C, one pertaining to the non-radiality of the mechanical loading and the other to a temperature history effect.

### 3.4 Anisothermal tests

Several real anisothermal test have been performed in order to confirm the results presented above and to compare them to the numerical predictions given by the behavior model [21-22] [25]. The problems of thermal dilatation are avoided by performing anisothermal torsion tests since the axial strain is not inhibited and the shear strain can be controlled by the temperature signal via an extensometer and a micro-computer. Tests are performed in phase, in 1/4 phase, in phase opposition, in 3/4 phase and in double phase (two mechanical cycles for a thermal cycle), for three successively increasing strain levels (around 40 cycles at each level). The results of the in phase and 1/4 phase tests are shown in Fig. 8 and 9 for the last cycles at each strain level.

Since no shear dilatation exists, the tests in opposition and 3/4 phase shift can be deduced from the preceeding ones (outside of the first quarter cycle) by symmetry with respect to the origin. If the stress levels ($\Delta\sigma/2$) are compared at the characteristic points of the cycle (inversion and singular points), where the strain and temperature are known for the same conditions under isothermal loading, then it can be shown that a difference of about 70 MPa (in equivalent) exists for the anisothermal loadings. These results confirm the conclusions drawn for stepwise isothermal experiments.

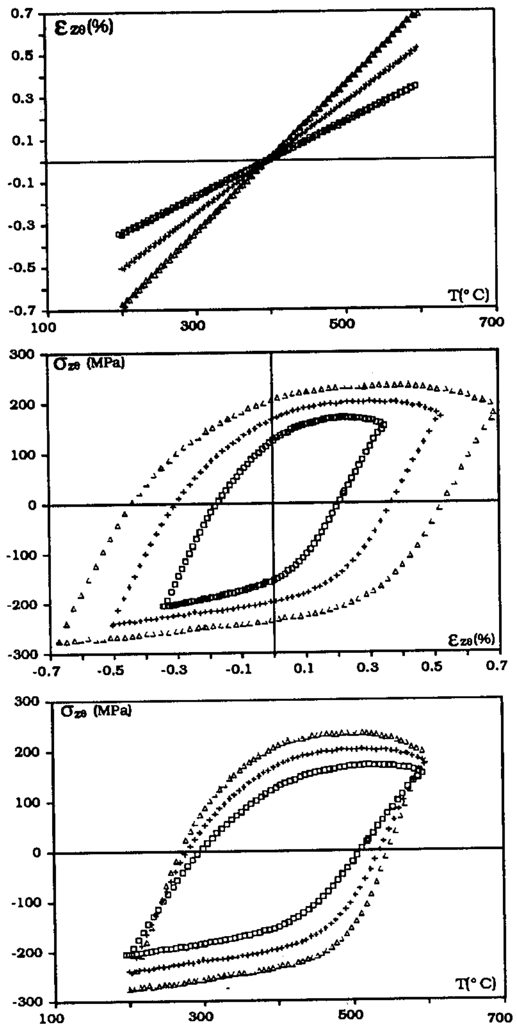

Fig. 8 : Anisothermal in phase torsion test.

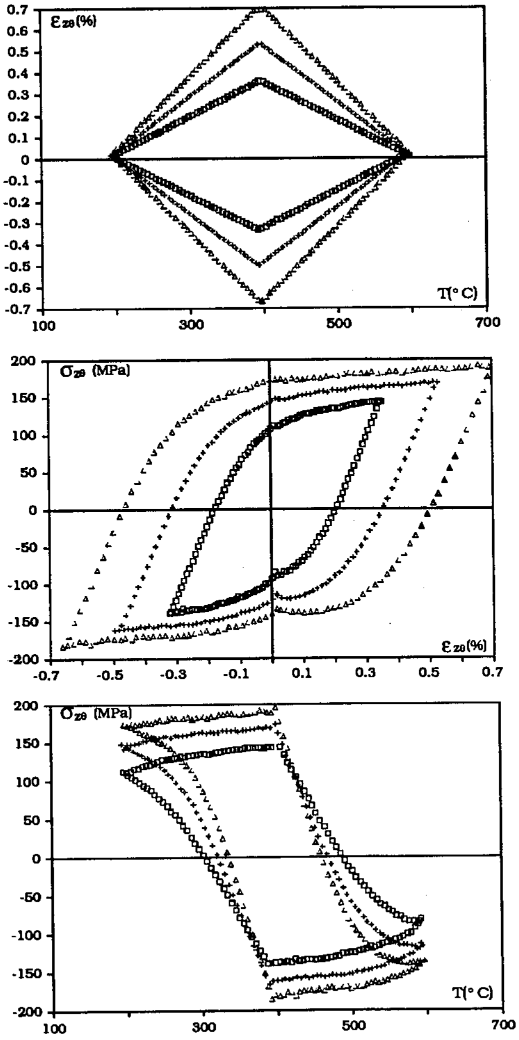

Fig. 9 : Anisothermal 1/4 phase torsion test.

## 4 - DISCUSSION

The existence of a temperature history effect has been demonstrated and its characteristics described. However, its physical nature remains to be explained. This effect is directly related to the existence of a "hump" in the curve $\Delta\sigma/2 = f(T)$, creating a non admissible domain where $\partial(\Delta\sigma/2)/\partial T$ is positive. Note that in the absence of a maximum, for example in monotonic tests (Fig. 1) or for other solid solutions [21-22], this effect is absent. In other words, the physical nature of the temperature memory phenomena is related to physics of the hardening mechanism associated to this maximum.

Several studies [28-30] [24] clearly show that between 200 and 600°C there exist very strong interactions between dislocations and point defect configurations in these steels, creating this hardening at intermediate temperature [24] [28] [30]. This interaction phenomena is very sensitive to the loading rate and can lead to a negative coefficient of the stress sensitivity with respect to the rate, a fact which has already been observed for this steel [24-25] [30]. The amplitude of the maximum is thus a function of the strain rate and in all logic, the effect of the temperature history should also depend on the rate in the sense that the "hump" is more pronounced for smaller rates. This point has been verified experimentally. Moreover, since the interactions occur with the dislocations present in the material, thus independently of the activated slip planes which are a function of the non-radiality of the loading, it would seem logical to find this history effect in uniaxial as well as biaxial tests. It can be accepted that short distance pinning of the dislocations exists by the different point defect configurations and this pinning is permanent and increases in the temperature range of 300 to 550°C.

This mechanism, which is intrinsic to solid solutions, explains the memorization of the maximal temperature. Note that this type of hypothesis has already been suggested by several authors [4] [6] [13]. The limited scope of this article does not allow the details of the modelization to be described. It can simply be noted that the unified model developed by Delobelle et al. [21] [24-25] for isothermal conditions between 20 and 650°C and describing the interaction phenomena observed between 200 and 600°C, is altogether able to describe to the totality of experiments presented above, assuming that the anisothermal formulation is used (appearance of terms in $(1/X)(\partial X/\partial T)\dot{T}$) and the integration of a small module allowing the memorization of $T_{max}$ to be described when $\partial(\Delta\sigma/2)/\partial T > 0$.

## 5 - CONCLUSIONS

With the aid of uniaxial and biaxial ($\phi$ = 0 and 90°) stepwise isothermal tests, it is shown that this austenitic steel possesses a temperature history phenomena which is independent regardless of the type of loading and is intrinsically related to a solid solution effect. The material memorizes the maximal temperature attained in the domain where the derivative of the maximal cyclic stress with respect to the temperature is positive. Real anisothermal tests, where mechanical and thermal loadings evolve simultaneously, have confirmed this observation.

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