

# Hysteretic behaviour of a Cu–Zn–Al single crystal during superelastic shear deformation

L. Orgéas<sup>a,\*</sup>, A. Vivet<sup>b,c</sup>, D. Favier<sup>a</sup>, C. LExcellent<sup>b</sup>, Y. Liu<sup>d</sup>

<sup>a</sup> *Laboratoire Sols-Solides-Structures, CNRS—Universités de Grenoble, B.P. 53, 38041 Grenoble Cedex 9, France*

<sup>b</sup> *Laboratoire de Mécanique Appliquée R. Chaleat, CNRS—Inst. des Microtech. de Franche Comté—Université de Franche Comté, 25000 Besançon, France*

<sup>c</sup> *Laboratoire Universitaire de Recherche Scientifique d'Alençon, Université de Caen Basse-Normandie—IUT d'Alençon, 61250 Damigny, France*

<sup>d</sup> *Department of Mechanical and Materials Engineering, University of Western Australia, Crawley, WA 6009, Australia*

Received 20 February 2004; received in revised form 16 April 2004; accepted 28 April 2004

Available online 20 May 2004

## Abstract

The present work investigates the hysteretic behaviour of Cu–Zn–Al single crystal submitted to superelastic shear tests. Major symmetric loops were performed within the shear strain limit of the stress-induced martensitic transformation. Minor loops inside the major loops allowed to analyse more closely the hysteretic behaviour of the Cu–Zn–Al single crystal. Experimental results obey most of the phenomenological observations previously established for polycrystalline Ni–Ti shape memory alloys.

© 2004 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

**Keywords:** Shape memory alloys; Single crystal; Simple shear tests; Superelasticity; Hysteresis

## 1. Introduction

Stress-induced thermoelastic martensitic transformation, thermally-induced thermoelastic martensitic transformation, as well as martensite reorientation occurring during thermomechanical loading of Shape Memory Alloys (SMAs) are not perfectly reversible physical processes: they always exhibit mechanical or thermal hysteresis, even at low loading rate. Austenite/martensite or martensite/martensite interfaces, as well as internal faults such as grain boundaries (in the case of polycrystalline SMAs), precipitates, or dislocations restraining the movement of transformation-phase boundaries are often believed to be responsible for the hysteresis observed in SMA [1]. A large number of experimental studies have been devoted to the analysis of the complex hysteretic behaviour of polycrystalline or single crystal SMAs (Cu-based or NiTi-based alloys). They focused on hysteresis occurring during thermally-induced martensitic transformation [2–4], stress-induced

martensitic transformation in superelasticity [5–10], and martensite reorientation in ferroelasticity [10,12]. A set of phenomenological observations describing the hysteresis have been deduced from such experimental results [10,11]. These typical observations have to be accounted for in effort to model the thermomechanical behaviour of SMAs, since many SMA components are subjected to cyclic loading conditions. To further the current understanding of hysteresis in SMAs, the above phenomenological rules are reinvestigated in the case of the cyclic shear superelastic deformation of a Cu–Zn–Al single crystal. The shear loading was chosen for the reasons listed below:

- (i) The mechanical hysteretic behaviour of SMA single crystals has always been studied in tension since this test is relatively easily and quickly performed [11,12]. However, depending both on the geometry of the sample and on the intrinsic properties of the tested SMA [13], undesirable Lüders-like behaviour may occur [14–17]. In that case, the global deformation of the sample occurs with the propagation of localized deformation bands and corresponding tensile engineering stress–strain curves exhibit a well-known horizontal plateau. Conclusions drawn

\* Corresponding author. Tel.: +33-4-76-82-70-73; fax: +33-4-76-82-70-43.

E-mail address: [laurent.orgeas@hmg.inpg.fr](mailto:laurent.orgeas@hmg.inpg.fr) (L. Orgéas).

from tensile experiments showing such a heterogeneous deformation mode are believed to be artificial and irrelevant for the study of mechanical hysteresis of SMAs [18]. It is believed that the shear deformation mode is efficient to prevent the development of localized deformation. For example, it has been shown that shear superelastic deformation of polycrystalline Ni–Ti alloys was macroscopically uniform when localized deformation could be observed in tension for the same alloy [16].

- (ii) The adoption of simple shear as the deformation mode also allows mechanical cycling to be performed symmetrically about the zero strain point: it has been shown that centred cycling about the zero strain point led to a minimization of undesirable martensite stabilization effects [10].
- (iii) To the best of our knowledge, no experimental results have been reported in the literature concerning cyclic superelastic shear deformation of a SMA single crystal, even though the macroscopic simple shear deformation mode is very close to the deformation observed at the martensite variant level: martensitic transformation as well as martensite reorientation are mainly deviatoric and display very small volume change [19].

## 2. Experimental procedure

Experiments performed in this work have been carried out on the same parallelepiped Cu–23.73Zn–9.4Al (at%) single crystal that was already deformed in a previous work [20]. Its processing is detailed in [21]. Its lattice parameters, orientation with respect to the loading axis, and characteristic temperatures of free-stress forward and reverse martensitic transformations are given in [20]. The single crystal was sheared with a simple shear device mounted on an Adamel-MTS DY35 universal mechanical testing machine using a load cell of 20 kN [22]. The apparatus was equipped with a silicon oil bath for temperature control so that the specimen was heated to an accurate constant temperature of  $333 \pm 0.1$  K ( $\approx 35$  K above  $A_f$ ). The sample was sheared at a very low shear rate  $\dot{\gamma}$  of  $1.7 \times 10^{-3}$  s $^{-1}$ . The thermally-induced effects due to the release and absorption of the latent heat associated with stress-induced martensitic transformation are known to be negligible for tests conducted in a liquid bath and for such low shear rate values: the testing condition could be considered practically isothermal.

The single crystal was first cycled symmetrically about the zero shear strain position with a shear strain magnitude  $\Delta\gamma_M$  of 40% for 50 cycles, in order to get a stabilized and highly repeatable superelastic stress–strain behaviour: the magnitude of the shear strain  $\Delta\gamma_M$  was chosen so that the forward stress-induced mar-

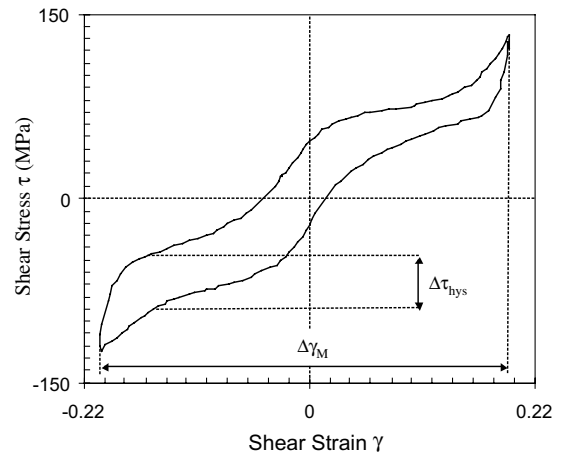
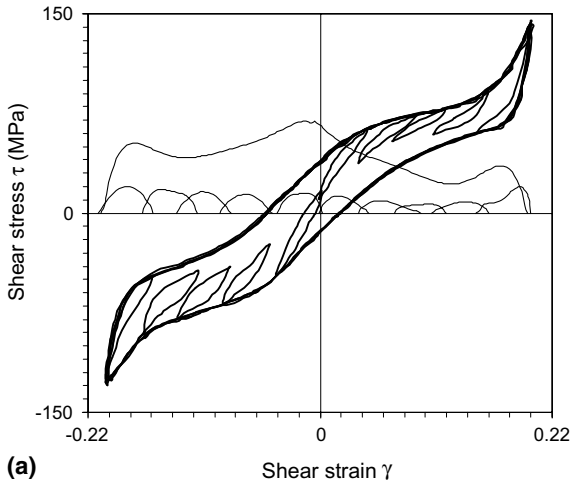


Fig. 1. Major loop between  $\gamma = -20\%$  and  $\gamma = 20\%$  ( $\Delta\gamma_M = 40\%$ ).

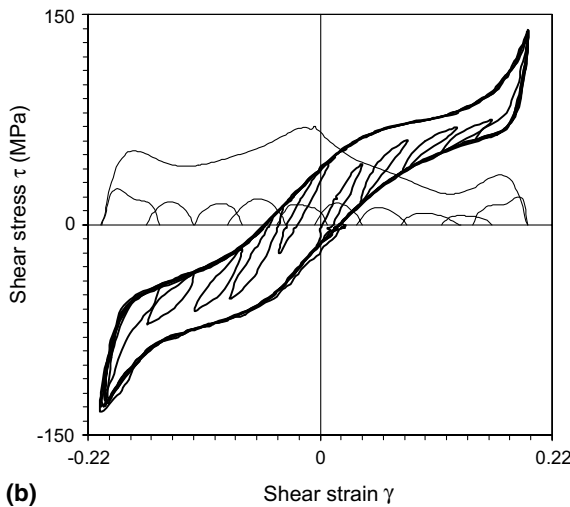
tenstic transformations could be considered as almost complete in both shear directions (see Fig. 1). Specially designed mechanical sequences made of centred major loops ( $\Delta\gamma_M = 40\%$ ) and minor loops (partial forward and reverse transformations) were then applied to the sample to study its hysteretic response. After each minor loop, the single crystal was systematically submitted to a centred major loop in order to restrain martensite stabilization [10].

## 3. Results

Fig. 2 shows a collection of internal loops performed inside the centred major loop of  $\Delta\gamma_M = 40\%$ . These minor loops of strain magnitude  $\Delta\gamma_m = 4\%$  were performed with different initial starting points “ $r$ ”. Minor loops were realized during stress-induced forward transformation (Fig. 2a) and stress-restrained martensitic transformation (Fig. 2b). It is clearly seen that each minor loop is closed at its starting point  $r(\tau^r, \gamma^r)$ , and is enclosed inside the major loop. The thin curves also plotted in these figures represent the stress hysteresis  $\Delta\tau_{hys}$  associated to the major loop and to all minor loops, defined as the difference of stress between the forward and reverse transformations at any given strain of the considered loop (see Fig. 1). Fig. 3 indicates that  $\Delta\tau_{hys}$  of a minor loop performed between  $\gamma^r$  and  $\gamma^r - \Delta\gamma$  during forward transformation is identical to  $\Delta\tau_{hys}$  of the minor loop performed between  $\gamma^r - \Delta\gamma$  and  $\gamma^r$  but during reverse transformation. This result is similar to the case of polycrystalline Ni–Ti [10]. However, results obtained for the Cu–Zn–Al single crystal is different from results obtained with the polycrystalline Ni–Ti in two aspects. Firstly, it is shown that  $\Delta\tau_{hys}$  is dependent on the shear direction, i.e., the sign of  $\gamma$ ,  $\Delta\tau_{hys}$  being larger when  $\gamma < 0$ . This effect is observed for the major loop and all the minor loops. The average values



(a)



(b)

Fig. 2. Subloops with  $\Delta\gamma = 0.04$  performed inside the major loop, and with different starting points  $(\tau^r, \gamma^r)$  located during forward stress-induced transformation (a), stress-restrained transformation (b). Bold curves: stress-strain curves. Thin curves: stress hysteresis magnitude  $\Delta\tau_{\text{hys}}$ .

$\Delta\tau_{\text{hysmax}}$  of the maximum difference of stress between the forward and reverse transformations at any given strain of a minor loop are approximately 20 MPa when  $\gamma < 0$  and 14 MPa when  $\gamma > 0$ . Secondly, the bold curves reveal that  $\Delta\tau_{\text{hys}}$  is a function of  $\gamma^r$ . This dependence is mainly observed for minor loops starting either in the vicinity of the zero strain point or at the highest shear strain of the major loop.

Fig. 4 illustrates the behaviour of the single crystal submitted to minor loops of identical starting point  $(\tau^r, \gamma^r)$  but with different strain magnitudes  $\Delta\gamma_m$ . The starting points  $r$  of each minor loop were  $(\tau^r = \pm\tau_{\text{max}}, \gamma^r = \pm\gamma_{\text{max}} = \pm 20\%)$  and  $(\tau^r = 0, \gamma^r)$  in Fig. 4a and b, respectively. It is shown that the stress hysteresis is an increasing function of the strain magnitude  $\Delta\gamma_m$ .

Similarly to the case of polycrystalline Ni-Ti [10], a complex cyclic loading was performed in order to better

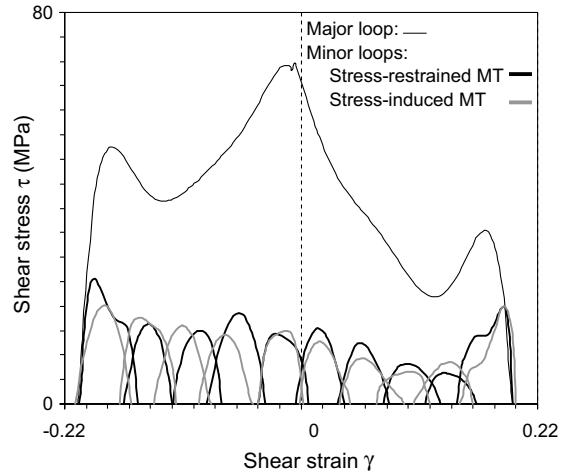
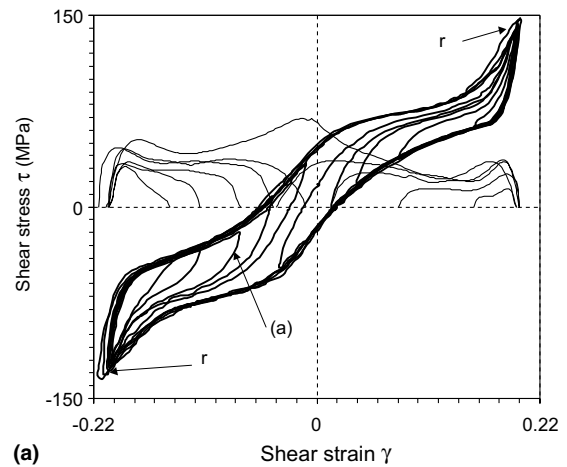
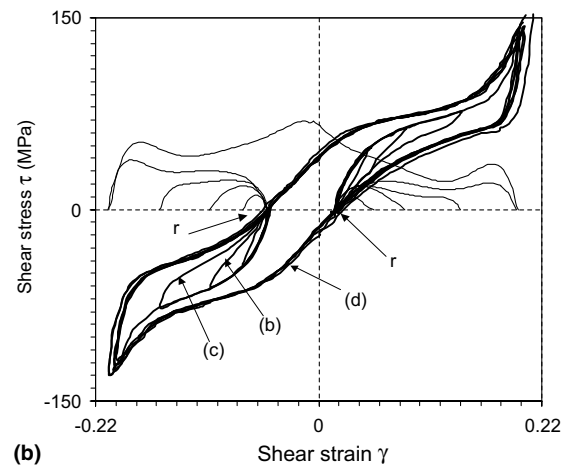


Fig. 3. Stress hysteresis  $\Delta\tau_{\text{hys}}$  of the major and minor loops performed in Fig. 2a (stress-induced forward martensitic transformation, grey bold curves) and Fig. 2b (stress-restrained martensitic transformation, black bold curves).



(a)



(b)

Fig. 4. Subloops with increasing the shear strain magnitude  $\Delta\gamma$ , performed at a starting point  $(\tau^r, \gamma^r)$  of  $(\tau_{\text{max}}, \gamma_{\text{max}})$  (a), of  $(0, \gamma^r)$  (b). Bold curves: stress-strain curves. Thin curves: stress hysteresis magnitude  $\Delta\tau_{\text{hys}}$ .

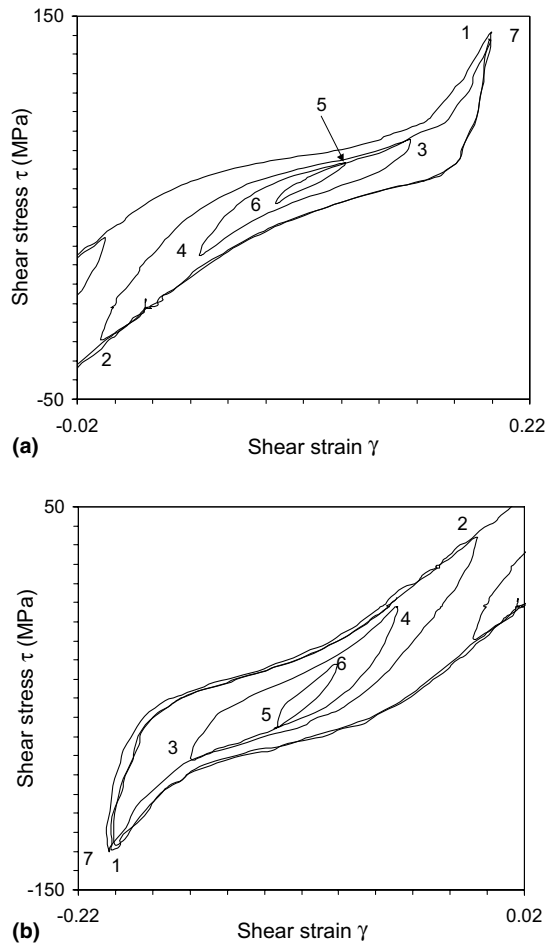


Fig. 5. Discrete memory concept when  $\gamma > 0$  (a) and  $\gamma < 0$  (b).

underline the role of the previous deformation history on the hysteretic behaviour of the single crystal. Hence, a special loop cycling depicted in Fig. 5, was applied to the superelastic Cu–Zn–Al single crystal. This loop cycling started at the maximum strain of the major loop (point 1 for which  $\tau^1 = \pm\tau_{\max}$  and  $\gamma^1 = \pm\gamma_{\max}$ ) and continued following the numerical order of the return points marked in the figures. Fig. 5a represents the single crystal’s response to this particular cycling when  $\gamma > 0$ , and Fig. 5b when  $\gamma < 0$ . In both cases, it was observed that during the section {6–7}, the stress–strain curves passed successively through all the previous starting points, i.e., 5, 3 and 1, closing all the incomplete partial loops. The re-assembling of the starting points along the {6–7} path underlines the role of the previous deformation history on the hysteretic behaviour of the single crystal. This will be discussed in the next section.

#### 4. Discussion

A set of typical observations describing the hysteresis in SMA have been deduced from experimental results

obtained with polycrystalline Ni–Ti alloys. These experimental evidences have been established for hysteresis occurring during homogeneous deformation associated either with martensitic transformation [10,11] or martensite reorientation [10]. The experimental results exposed above allow us to reinvestigate these typical observations in the case of a Cu–Zn–Al single crystal submitted to simple shear superelastic deformation:

- (i) Similarly to the case of polycrystalline SMAs, the stress hystereses  $\Delta\tau_{\text{hys}}$  of a minor loop of strain magnitude  $\Delta\gamma_m$  are identical during forward and reverse stress-induced martensitic transformation, as shown in Fig. 3.
- (ii) The magnitude of hysteresis stress  $\Delta\tau_{\text{hysmax}}$  increases with the strain amplitude of the minor loop  $\Delta\gamma_m$  as shown in Fig. 4.
- (iii) A minor loop initiated at a given starting point  $r(\tau^r, \gamma^r)$  always closes at this starting point. This is illustrated in Figs. 2 and 4. As an example, for the minor loops started at  $\gamma = -\gamma_{\max}$  (Fig. 4a), the first branch of the minor loops coincides with the unloading part of the major loop. However, when the loading is reverted (for example curve (a)), the minor loops leaves the major loop and reaches it again only at their common starting and closure point  $\gamma = -\gamma_{\max}$ . The role of the starting point remains meaningful when the minor loop is initiated elsewhere, as evident in Fig. 4b. These starting points (marked as “r” in Fig. 4a and b) are similar to the reference points defined by Gu elin [23].
- (iv) The previous observation is supported by considering the complex cycling of Fig. 5. In this figure, and whatever the sign of  $\gamma$ , point 6 is reached reverting successively the imposed strain at points 1, 2, 3, 4 and 5. Hence, minor loop {1–2–7} (or {1–2–1}) is seen as the parent loop of the minor loop {3–4–3}, which is in turn seen as the parent loop of the minor loop {5–6–5}. Following (iii), minor loop {5–6–5} is closed at its starting point 5. Furthermore, return points 3 and 1 are reached successively when increasing the imposed strain from points 6 to 7. This behaviour suggests that the material reacts as it has memorized particular points during the {1–2–3–4–5} trajectory. Two important remarks are drawn from the last observation. Firstly, during a complex cycling, all the starting points  $(\tau^r, \gamma^r)$  of the incomplete minor loops are memorized. Secondly, once a minor loop is closed, its starting point is forgotten and the material behaviour is identical to what it would have been if the minor loop had not been performed. The term of erasable micromemory [5] has been introduced to characterize this behaviour: the parent loop is not affected by all the minor loops performed inside it.

- (v) Rules (iii) and (iv) imply that the relevant reference point ( $\tau^r$ ,  $\gamma^r$ ) can change during a complex loading, similar to those presented in Fig. 5. As an example, during the {6–7} branch, the relevant reference point is successively ( $\tau^6$ ,  $\gamma^6$ ), then ( $\tau^4$ ,  $\gamma^4$ ) and ( $\tau^2$ ,  $\gamma^2$ ) along the loading arches {6–5}, {5–3} and {3–7} (or {3–1}), respectively. The choice of the relevant reference point between all the previous starting point is function of the loading path and is similar to the discrete memory concept [23].
- (vi) If a minor loop is performed inside a parent loop, it must be inside the parent loop. This is a direct consequence of (ii) and (iii). For instance, minor loop (a) (for which  $\Delta\gamma_m = 12\%$ ) of Fig. 4a has a similar starting point of the centred major loop (for which  $\Delta\gamma_m = \pm 20\%$ ), i.e.,  $(-\tau_{\max}, -\gamma_{\max})$ . Hence, according to (iii), the first branches of the major loop and minor loop (a) must be identical: this is obvious in Fig. 4a. Following (ii), the stress hysteresis magnitude of minor loop (a) must be lower than the major loop's one. Thus, the minor loop (a) must be inside the major loop which is its parent loop: this is confirmed in Fig. 2a. A similar reasoning is valid with minor loop (b) of Fig. 4b, which is inside minor loop (c), which in turn is inside the major loop (d).
- The previous experimental evidences have been already established for polycrystalline SMAs. Results obtained in the present paper show that they are still valid for homogeneous superelastic deformation of a Cu–Zn–Al single crystal. Nevertheless, specific observations have to be added in the case of our experiments on the Cu–Zn–Al single crystal:
- (vii) The stress hysteresis magnitude  $\Delta\tau_{\text{hys}}$  is a function of the sign of  $\gamma$ . As evident in Figs. 2–4,  $\Delta\tau_{\text{hys}}$  was always higher when  $\gamma < 0$ . This was not observed in the case of non-textured polycrystalline NiTi [10]. As it was previously proposed for shear transformation stresses and strains [20], the observed asymmetric hysteretic behaviour may also be ascribed to the low crystallographic symmetry of the Cu–Zn–Al single crystal.
- (viii) For polycrystalline SMAs, it has been established that the magnitude of the stress hysteresis of a loop is independent of the pre-deformation, both for ferroelastic and superelastic behaviour. As shown in Fig. 3, the stress hysteresis of the minor loops performed between 8% and 12% on one hand and between 12% and 16% on the other hand are almost identical. That obeys the previous rule established for polycrystalline SMAs.
- (ix) However, looking at the stress hysteresis magnitude  $\Delta\tau_{\text{hys}}$  of the minor loops performed between 16% and 20%, it is seen that  $\Delta\tau_{\text{hys}}$  is higher compared to the previous ones. This could be

explained by the high values of the shear deformation for which the martensitic stress-induced transformation is likely to be almost complete. The deformation is suspected to be the result of two superimposed mechanisms: (1) further stress-induced martensitic transformation of some residual austenite and (2) reorientation of some martensite variants induced either by the previous shear deformation or by the gripping of the plate between the pressure grips of the shear device.

- (x) The stress hysteresis magnitude of the minor loops performed for low values of shear stress, at the vicinity of the zero strain (i.e., minor loops between 0% and 4% and between 4% and 8% plotted in Fig. 3) is also dependent on the shear strain. The stress-restrained martensitic transformations during unloading of the major loops are indeed not complete, which leads to residual deformation at zero stress. For low values of shear stress, during loading, the deformation is likely to be the result of two superimposed mechanisms: (1) direct stress-induced forward martensitic transformation in the shear direction of some austenite obtained by stress-restrained transformation during unloading and (2) reorientation of some residual martensite variants induced by the shear deformation in the opposite direction and not transformed back in austenite during unloading. This could explain the gradual evolution of the stress hysteresis magnitude  $\Delta\tau_{\text{hys}}$  observed for  $\gamma > 0$  to the stress hysteresis magnitude observed for  $\gamma < 0$ .

## 5. Conclusion

Many experimental studies have been devoted to a better understanding of hysteresis occurring during thermomechanical loading of single or poly SMA crystals. All these works led to a common set of phenomenological observations governing the hysteretic behaviour of these materials in the case of low thermomechanical loading rates. The present work focused on the simple shear hysteretic behaviour of a superelastic Cu–Zn–Al single crystal. The above observations have been confirmed and completed in the present paper. It appears that most of the established rules do not depend on the deformation mode (stress-induced martensitic transformation, thermally-induced martensitic transformation, and martensite reorientation), or on the studied material (NiTi-based alloys, Cu-based alloys, etc.), or on its microstructure (poly or single crystals). In that sense, they have an universal character. These experimental evidences must be accounted for in the effort to model the thermomechanical behaviour of SMAs.

**References**

- [1] Delaey L, Aernoudt E. Proceedings of ICOMAT 86. JIM . 1986. p. 926.
- [2] Planes A, Castan T, Ortin J, Delaey L. J Appl Phys 1989;66:2342.
- [3] Cornelis I, Wayman CM. Scripta Metall 1976;10:359.
- [4] Amengual A, Cesari E, Segui C. Proceedings of ICOMAT 79, Monterey, USA. 1992. p. 377.
- [5] Verguts H, Delaey L, Aernoudt E, Vermeesch W. Proceedings of Euromech, Warszawa, Poland. 1987.
- [6] Lü L, Aernoudt E, Delaey L. Scripta Metall 1988;22:1435.
- [7] Ortin J. J Appl Phys 1992;71(3):1454.
- [8] Fu S, Huo Y, Müller I. Acta Mech 1993;99:1.
- [9] Tourabi A, Wack B, Favier D. Mater Sci Forum 1990;56:535.
- [10] Orgéas L, Liu Y, Favier D. J Phys IV 1997;7(C5):477.
- [11] Cory JS, McNichols Jr JL. J Appl Phys 1985;58(9):3282.
- [12] Müller I, Zak G. J Phys III 1995;5(C8):197.
- [13] Liu Y, Houver I, Xiang H, Bataillard L, Miyazaki S. Metall Mater Trans 1999;A30:1275.
- [14] Miyazaki S, Imai T, Otsuka K, Suzuki Y. Scripta Metall 1981;15:853.
- [15] Shaw JA, Kyriakides S. J Mech Phys Solids 1995;43(8):1243.
- [16] Orgéas L, Favier D. Acta Mater 1998;46(15):5579.
- [17] Liu Y. Mater Sci Eng 1999;A271:506.
- [18] Orgéas L, Favier D. J Phys IV 2001;11:67.
- [19] Caneiro A, Chandrasekaran M. Scripta Mater 1988;22:1047.
- [20] Vivet A, Orgéas L, LExcellent C, Favier D, Bernardini J. Scripta Mater 2001;41:33.
- [21] LExcellent C, Goo BC, Sun QP, Bernardini J. Acta Mater 1996;44(9):3773.
- [22] Manach PY, Favier D. Mater Sci Eng A 1997;A222:45.
- [23] Guélin P. J Mécanique 1980;19(2):217.