THERMODYNAMICS AND HYSTERESIS BEHAVIOUR OF THERMOELASTIC MARTENSITIC TRANSFORMATIONS

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Abstract - This work reviews different approaches to the thermodynamics of the martensitic transformation in shape-memory alloys developed in the last years, and attempts to give a general unified version in terms of an energy balance which not only considers equilibrium situations but includes as well the possibility of dissipative effects. The thermoelastic transformation is studied in a quasistatic continuous formalism which results from the assumption that the characteristic relaxation times between thermoelastic equilibrium states are comparatively negligible. The analysis reveals the role of different energy contributions (chemical, elastic and dissipative) in the transformation, and the possibility of their evaluation from calorimetric measurements. Concerning hysteresis, we review the memory properties of partial cycles common to this whole class of systems, and present a number of recent approaches developed to model these properties.

1.- Introduction.

A realistic study of the thermodynamics of thermoelastic martensitic transitions is compelled to take into account characteristic features of these transitions [1,2] such as: (a) surface energy contributions associated with the interfaces separating the high (H) and low (L) temperature phases and different variants of the L phase, (b) elastic strain energy contributions related to the accommodation of domains of the martensitic phase within the H phase matrix and (c) dissipative effects decisive for the understanding of the peculiar features of hysteresis cycles in these materials. The main difficulty in dealing with these contributions is their heterogeneous character. The situation is even more complex for the dissipative effects since the precise operative mechanisms are still not well identified.

This work is oriented towards a thermodynamic study of the transition, which starts from a general energy balance between the contributions listed and whose purpose is to stablish relations between global quantities of the transition. In this sense, this type of approach does not require to detail expressions for the different contributions.

The thermoelastic martensitic transition is described as a succession of metastable states. The paper concentrates on the assumptions behind this type of description and on the experimental results supporting them. Though we give a number of explicit results derived from the formulation, additional details and results for situations not treated here can be found in the references given.

2.- Thermodynamics: general framework.

We begin by considering a thermally-induced martensitic transition with no applied stress. The L phase is obtained from the H phase essentially by a homogeneous shear accompanied by a very small

volume change. We must consider that, as a consequence of the symmetry properties of the H phase, the L phase is g times degenerated: g structures (variants) of the L phase can appear, differing only in their relative crystallographic orientation, but energetically equivalent in the absence of any external stress field. Usually, in the absence of external field, the L phase variants form in self-accommodating groups minimizing the internal strain fields and hence the bulk elastic strain energy in the system [3,4].

During growth, different mechanisms can give rise to dissipative effects. The precise mechanisms at the origin of energy dissipation are still controversial and, moreover, have been little studied either experimentally or theoretically. In any case, it seems well stablished [5,6] that one of the dominant mechanisms of hysteresis in thermally-induced transformations is the relaxation of elastic strain energy. Indeed, the elastic energy stored in the system, which increases in a continuous way as an isolated single variant grows thermoelastically, can be partially relaxed when an ensemble of variants (two or more) join to form a self-accommodating group or when the variant reaches a free surface of the system. This mechanism prevents an undefinite increase of the bulk elastic strain energy, which would finally lead to plastic accommodation [7].

Accurate observations reveal that the transformation proceeds through a series of discrete steps between metastable equilibrium states [8-10]. In each transformation step, elastic energy is stored in the system and, at the same time, energy is dissipated. For this reason, at the new metastable situation the temperature of the system must be changed to resume transformation. For low enough temperature rates, however, the dissipative effects (associated to each transformation jump) take place in time intervals much smaller than the times of appreciable variation of the driving force. Thus, the system spends the overwhelming majority of its time in a situation of thermoelastic equilibrium: practically at any temperature in the temperature interval where phases H and L coexist the system is in thermoelastic equilibrium. This has been checked by the observation that, when the system is kept at a constant temperature within the two-phase region, it does not show any tendency to transform further [8]. Concerning this point, however, we note that care must be taken with other diffusive processes that might take place simultaneously with the transformation, and can mask the intrinsic features of the martensitic transition. In metallic alloys the most common is a process of atomic ordering; as an extreme example, it has been reported that Cu-Zn-Al alloys undergo a martensitic transition at constant temperature and no applied external stress, induced by an atomic ordering process [11]. These extrinsic, diffusive processes, can always be avoided by well defined heat treatments [12]. In what follows we will disregard these situations and assume always that the system is in thermoelastic balance in practically any state within the two-phase region, so that it is a good approximation to consider the transformation as progressing continuously and to study the thermodynamics of the transformation using a quasistatic formalism.

Some of the features described are altered when an external uniaxial stress is applied to the system. The stress field breaks the degeneracy and for a large enough stress only a limited number (usually just one) out of the g possible variants is induced. In ideal experimental conditions, when only one interface separates the two phases L and H, the transition takes place at a given temperature and applied stress. There is not elastic strain energy stored in the bulk, and the interfacial energy takes a constant minimum value because there is only one interface, of constant dimensions, in the system. Dissipation still takes place, though considerably minimized, since there is not elastic energy relaxation. This situation has been experimentally studied and modelled by Lovey et al. [13], who ascribe the energy dissipation to the interaction between the moving interface and dislocations of the crystal. The mechanism gives rise to a very small thermal hysteresis, of the order of 0.1 K.

3.- Thermodynamics: results.

Since the end of the 70's several formulations have been developed to deal with the thermodynamics of thermoelastic martensitic transitions. A number of these formulations are based on thermodynamic equilibrium formalisms, where energy dissipative processes are ignored, either homogeneous [14] or

taking into account local heterogeneities due to elastic effects [15]. An interesting approach, due to Cory and McNichols [16], was the first in considering the role of energy dissipation on the thermodynamics of shape-memory alloys. The origin of dissipation is not discussed, but the authors emphasize that the standard non-equilibrium formalisms, of application to non-quasistatic relaxation phenomena, are not suitable to study non-relaxation hysteretic processes. The distinction between non-quasistatic and hysteretic phenomena comes from the difference in their characteristic time scales, as discussed in the previous section [17].

Recently, we suggested a formulation which integrates these ideas and can be considered a generalization of the previous approaches. Firstly we conjectured that all the energy losses could be considered as mechanical work that the system dissipates without entropy production. This possibility is consistent with the idea that dissipation is mainly due to elastic energy relaxation during transformation and with the fact that elastic energy can be released in the form of elastic waves. The fact that acoustic waves are detected as acoustic emission in the transformation [9] validates the argument. Obviously, once these elastic waves are absorbed in the neighbourhood of the system considered, the dissipative work gives rise finally to entropy production. Experimental results of calorimetric measurements on thermally-induced transformations, however, have revealed that entropy production is indeed very small and its contribution to energy dissipation is negligible.

This general formalism is established from a balance equation representing a general writing of the thermodynamic fundamental equation. The variables suitable to describe the system in the coexistence region of phases L and H are assumed to be independent of time, as follows from our previous discussion. Within this assumption, the transformation path (defined as x=x(T), with x the martensitic molar fraction) is independent of the variation rate of the driving force. Though there is some evidence of this static behaviour for low rates, there has not been yet a systematic study on this point. In copper based alloys, for example, preliminary experiments confirm that in thermally-induced transitions no noticeable changes of the transformation path occur for temperature rates varying in the range from 0.01 to 1 $Kmin^{-1}$ [18]. The behaviour for larger temperature rates, however, is yet to be studied.

The energy balance reads:

$$dU = TdS - T\delta S_i - dE_{el} + V_0 \sigma_{ij} d\varepsilon_{ij} - pdV - \delta W_i \qquad (1)$$

where U, S and V are internal energy, entropy and molar volume of the two-phase system, T is the temperature (assumed to be homogeneous) and p the hydrostatic pressure. $V_0 \sigma_{ij} d\varepsilon_{ij}$ is the mechanical work performed by external forces, which in the following we will assume to be uniaxial. dE_{el} is the stored elastic strain energy, a term that includes surface and volume contributions. δS_i is the entropy production and δW_i is the internal work dissipated in an irreversible way in forms other than heat; these two dissipative terms are defined to be non-negative, and since they are not exact differentials we use the symbol δ to express their infinitesimal variation.

It is interesting to introduce the thermodynamic potential H, called enthalpy, defined by:

$$H = U + pV \qquad (2)$$

Now the fundamental equation reads:

$$dH = TdS - T\delta S_i - dE_{el} + V_0 \sigma d\varepsilon + Vdp - \delta W_i \qquad (3)$$

In the following, to simplify the resulting equations, we will neglect possible differences in the heat capacity and elastic moduli of the two phases. This approximation is equivalent to state that the change in thermodynamic potentials in the transition is independent of temperature and applied stress.

For a hypothetical transition that would take place in equilibrium (ideal thermoelastic behaviour) we would have that $\delta S_i = \delta W_i = 0$. If in addition we disregard the elastic interaction

between the two phases we also have that $dE_{el}=0$ and then, at constant pressure:

$$Q = T_0(\sigma)\Delta S = \Delta H - V_0\sigma\Delta\varepsilon \qquad (4)$$

where Q is the heat exchanged and $T_0(\sigma)$ is the temperature at which the transition takes place in equilibrium under an applied stress σ. Equation (4) has come out to be suitable to interpret calorimetric results of thermally-induced thermoelastic martensitic transition of single crystals subjected to simple uniaxial stresses [19,20].

In particular, when $\sigma=0$:

$$\Delta H = T_0\Delta S \qquad (5)$$

where now $T_0=T_0(0)$. In addition, an ideal in-equilibrium formulation as represented by equation (4) gives the variation of the equilibrium temperature T_0 with uniaxial stress σ, i.e. the Clausius-Clapeyron equation:

$$\frac{dT_0}{d\sigma} = -V_0\frac{\Delta\varepsilon}{\Delta S} \qquad (6)$$

3.1 Thermally-induced transition.

For a thermally-induced transition without applied stress, $\sigma=0$ but $dE_{el}\neq0$. Then, in ideal equilibrium at constant pressure:

$$Q = \int_{T_s}^{T_f} TdS = \Delta H + E_{el} \qquad (7)$$

The integral must be performed along the transition path, between the hypothetical initial and final equilibrium temperatures T_s and T_f. Note the parallelism between equations (4) and (7). It is worth mentioning that the heat exchanged Q (the heat measured in a calorimeter) does not coincide with the enthalpy change ΔH (latent heat of transformation), due to the storage of elastic energy in the system.

Let us now consider the general case of a thermally-induced transition in a system subjected to uniaxial stress, including dissipative effects. Considering a quasistatic path (in the sense discussed previously), equation (3) leads to:

$$Q = \int_{M_s(\sigma),A_s(\sigma)}^{M_f(\sigma),A_f(\sigma)} T(dS-\delta S_i) = \Delta H + E_{el} + W_i \qquad (8)$$

Here, the integrals are computed between $M_s(\sigma)$ and $M_f(\sigma)$ for the H→L transition and between $A_s(\sigma)$ and $A_f(\sigma)$ for the L→H transition. The different contributions (ΔH, E_{el}, W_i and S_i) to this energy balance equation can be evaluated from the exchanged heat Q, measured in a calorimeter [21]. For a complete cycle, we get:

$$\oint \delta Q = \oint T(dS-\delta S_i) = \oint \delta W_i \qquad (9)$$

This procedure relies on the fact that, after a complete cycle, the system has come back to the same thermodynamic state from which the cycle started, and hence all the state functions return to their original values. This corresponds to the study of a system that does not evolve from cycle to cycle. In addition, we simply divide the dissipative work W_i between forward and reverse transitions in two

equal parts. Then:

$$\oint \delta Q = 2W_i \qquad (10)$$

The simplest method to compute the stored elastic energy is to assume that:

$$\int T\delta S_i \ll W_i < Q \qquad (11)$$

and neglect subsequently the entropy production. From equation (8), this results in:

$$E_{el} = Q - \Delta H - W_i \qquad (12)$$

where ΔH is obtained from equation (5) as:

$$\Delta H = T_0 \int^t \frac{\delta Q}{T} \qquad (13)$$

Figure 1a shows schematically the relative importance of the different energy contributions calculated with this procedure, and Table I a collection of values calculated from calorimetric measurements on a number of copper-based alloy systems.
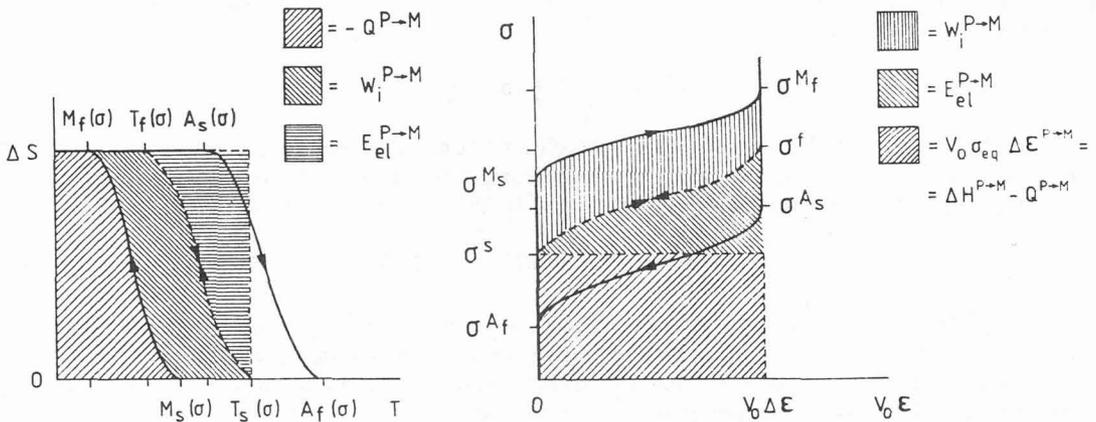

Figure 1.- Schematic representation of the hysteresis loop of (a) a thermally-induced transition under a constant applied stress σ, and (b) a stress-induced transition at constant temperature, showing the role of the different energy contributions in the forward transition, denoted here as P→M.

As an example of application, Picornell et al. [22] have used equation (10) to compare the hysteresis cycles of a Cu-Zn-Al crystal exposed to two different thermal treatments: air cooling or water quench from high temperatures. The main difference between the two cycles is in the nucleation and earliest stages of martensitic growth which, for the first of the treatments, take place much more abruptly and at lower temperatures. The energy dissipated in this explosive nucleation has been evaluated by comparing the areas of both transition cycles.

We have also developed more elaborated methods which allow for entropy production δS_i and for the possibility of a difference in heat capacity between phases H and L [23,24]. The results that follow from such extended formulation, however, confirm that the simplified treatment developed here is adequate to most practical purposes.

Table I. Summary of thermodynamic results, including the three energy contributions to the thermoelastic balance, for a number of copper-based alloy systems which undergo different kinds of martensitic transitions. Values are taken from references A: R.J.Salzbrenner and M.Cohen, Acta metall. **27**, 739 (1979) and B: J.Ortín and A.Planes, Acta metall. **36**, 1873 (1988).

Alloy composition	Transition	T_0	ΔT	Q^{H-L} J/mol	Q^{L-H} J/mol	ΔS^{H-L} J/molK	ΔS^{L-H} J/molK	E_{el} J/mol	W_i J/mol	$\mid \Delta H \mid$ J/mol
Cu27.7Al2.3Ni Single crystal Ref.A	$\beta \rightleftharpoons \gamma'$ Single interface	304	30	-515	536	-1.78	1.68	0	51	526
Idem Ref.A	$\beta \rightleftharpoons \gamma'$ multiple interface	299	30	-460	481	-1.67	1.58	42	48	526
Cu16.1Zn15.9Al Single crystal Ref.B	$\beta \rightleftharpoons \beta'$ multiple interface	238	5	-301	311	-1.32	1.33	15	8	320
Cu7.2Zn23.1Al Polycrystal Ref.B	$\beta \rightleftharpoons \gamma'$ multiple interface	249	28	-301	341	-1.41	1.42	25	29	350

3.2 Stress-induced transitions.

For a stress-induced transformation at constant temperature, in the pseudoelastic regime, equation (3) written for a complete cycle leads to:

$$\oint V_o \sigma de = T \oint \delta S_i + \oint \delta W_i \qquad (14)$$

showing that the energy dissipated in the cycle, either as entropy production or mechanical work, is given by the area of the cycle in a stress-strain diagram. The entropy production, in addition, can be determined now by measuring the heat exchanged by the specimen in the tensile cycle, because:

$$\oint \delta Q = \oint T(dS - \delta S_i) = -T \oint \delta S_i \qquad (15)$$

The overall process is exothermal, as the negative sign indicates. An experiment of this kind enables a direct verification of the hypothesis of null entropy production (see figure 1b). Sade et al. [25] have performed preliminary tests on Cu-Zn-Al single crystals and shown that the whole process is certainly exothermal, with energies dissipated as entropy production in the range from 5 to 10 % of the enthalpy change of the transition.

4.- General properties of the hysteresis cycle.

The incorporation of dissipative terms in the thermodynamic balance (3) has been motivated by the experimental fact that the martensitic transition, even in systems that are acknowledged to transform thermoelastically, displays hysteresis: when we change the sign of the driving force the system does not return via the same thermodynamic path.

A thermodynamic formulation incorporating dissipative effects enables: (a) evaluation of energy losses in the transition from the properties of the experimental hysteresis cycle, as shown in the previous section; (b) study of the evolution of energy losses with the transformed fraction along the transition path; and (c) characterization of the suitable functional dependence of the dissipative term in the energy balance, based on the particular information provided by the thermodynamic trajectories inside the two-phase region (partial hysteresis cycles). These two last items are developed in the following.

Consider a generalized Gibbs free energy defined by the following Legendre transformation:

$$G^* = H - TS - V_o \sigma \varepsilon \tag{16}$$

From equation (3) we arrive to:

$$dG^* = -SdT - dE_{el} - V_0 \varepsilon d\sigma + Vdp - T\delta S_i - \delta W_i \tag{17}$$

and hence:

$$dG^*|_{T,p,\sigma} = -dE_{el} - T\delta S_i - \delta W_i \tag{18}$$

Thus, at fixed T, p and σ, the change in G^* in our continuous formalism compensates for the changes in elastic and dissipative energies. When the martensite molar fraction x changes to x+dx we have:

$$dG^*|_{T,p,\sigma} = \left(\frac{\partial G^*}{\partial x} \right)_{T,p,\sigma} dx = (G_L^* - G_H^*)dx \tag{19}$$

which represents the driving force for the transition. Thus:

$$(G_L^* - G_H^*)dx = -\left(\frac{\partial E_{el}}{\partial x} \right)dx - \left(\frac{\delta E_{diss}}{dx} \right)dx \tag{20}$$

Here:

$$\delta E_{diss} = T\delta S_i + \delta W_i \geq 0 \tag{21}$$

independently of the sign of dx. Consequently, the thermoelastic balance equation (20) displays two types of branches: branches (+) associated with dx>0 (forward transition: H→L) and branches (-) associated with dx<0 (reverse transition: L→H).
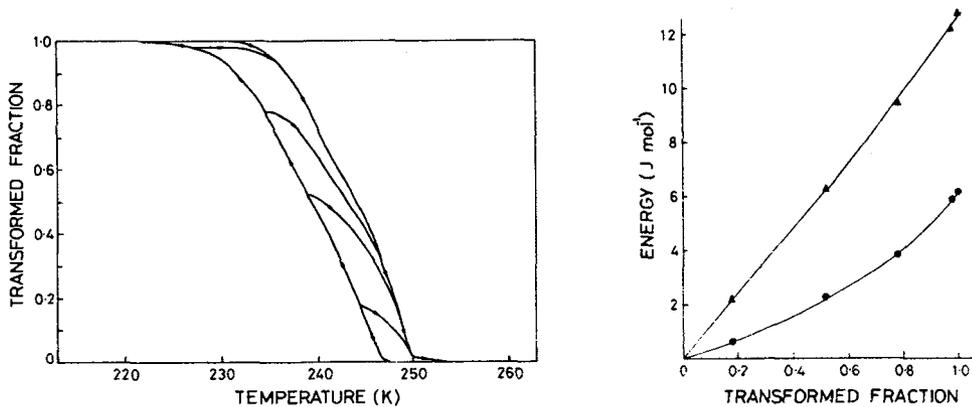

Figure 2.- A collection of partial hysteresis loops corresponding to the thermally-induced transition of a Cu-Zn-Al single crystal, obtained by calorimetry (a) and the associated evolution of dissipative energy (●) and reversible elastic energy (▲) as a function of the transformed fraction (b). From reference [26].

4.1 Evolution of E_{diss} with transformed fraction.

We have shown that the overall energy losses can be obtained as the area of the hysteresis cycle represented in the proper variables. This result also applies to partial cycles where the transition (forward or reverse) is interrupted at a transformed fraction x, the transition direction is reverted and the path continued until the initial state is reached again (figure 2a). Using this procedure, we have been able to determine the evolution with transformed fraction of the energy dissipated. In figure 2b we show the results obtained for the thermally-induced transition of a Cu-Zn-Al single crystal [26]. In this case the accumulated energy losses grow monotonously with transformed fraction up to a value representing about 5 % of the transition enthalpy.

4.2   Models for the dissipative energy and prediction of partial cycling features

The properties of thermodynamic paths inside the two-phase region are rather distinctive and require a special functional form of E_{diss} in equation (20). These properties [6,16,27] are summarized by:

(a)   All the transformation trajectories are located within a region of the x-σ-T space, the two-phase region, bounded by the trajectories of the complete cycle (x:0⇌1).

(b)   Each transformation trajectory depends only on the set of extreme values (return points) reached by the variable driving the transition (σ or T) along the trajectory. The system "keeps memory" of this set of values.

(c)   Each time that a transformation trajectory returns to an earlier extreme value of σ or T the influence of this value on the trajectory is lost (wiping out property). This important property makes the partial cycles to exhibit the self-similar behaviour represented schematically in figure 3.

The list above summarizes the intrinsic behaviour of partial cycles within the two-phase region, for which time plays no role other than a parameter. Actually, it represents an idealization of the experimental behaviour, valid in the quasistatic limit; we exclude the influence on the hysteresis cycles of relaxational phenomena such as ordering or stabilization and cycling effects. This behaviour has been observed in a variety of systems, including Cu-Zn, Cu-Zn-Al and Ni-Ti, for single crystals and policrystals, and for both thermally- and stress-induced transitions [26,28-31]. It is not strictly associated with the formation of self-accommodating domains of the low temperature phase, because it has been observed as well in single crystals under uniaxial stress, which transform to a single martensite variant [32]. Its microscopic origin, however, is yet unclear. For this reason, existing models of partial cycling behaviour are of a phenomenological nature. Moreover, it is interesting to mention that the same behaviour is also displayed by hysteresis loops in ferromagnetic materials [33].
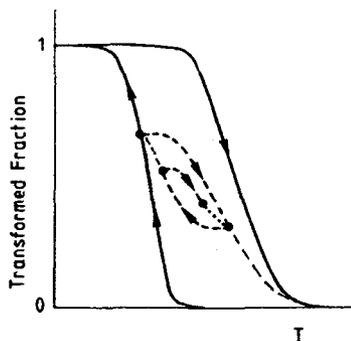

Figure 3.- Schematic behaviour of the transformation paths in a thermally-induced transition. Continuous lines represent a complete cycle which follows the boundary of the two-phase region. Broken lines represnt internal paths, and black points correspond to return points.

Müller and co-workers [34,35] have developed a Landau-Devonshire type of model for pseudoelastic hysteresis, in which the appearance of non-monotone load-deformation equilibrium curves gives rise to hysteresis. The width of hysteresis cycles is determined by the interfacial energy of the H and L phases, and internal hysteresis loops contain metastable states that loose their metastability on the line defining phase equilibrium. The model accounts for the existence of internal trajectories, but it does not seem to reproduce the memory properties (b) and (c) of the previous list.

Lü et al. [36] represent the system as a network of elements connected in parallel, i.e. subjected to the same strain. The balance equation (20) is verified in each element, but the driving force differs by a constant value from element to element. The dissipative term in each element is explicitly defined to keep memory of the last return point. Let us consider, for instance, a trajectory of the type (+); along this trajectory the dissipative term is written as a linear function of the increment in transformed fraction from the previous return point x^a:

$$\mu_1 + \mu_2(x - x^a) \tag{22}$$

We follow this trajectory (+) and, at x^b, we revert the sign of the driving force and start a trajectory (-) ending at x^c, where we return to the initial point x^b and close a partial cycle. Forcing the dissipative term at the return point x^b to be the same before and after the partial cycle results in:

$$\mu_2(x^b - x^a) = \mu_{2i}(x^b - x^c) \tag{23}$$

where μ_{2i} is the slope of the dissipative term in the (+) branch of the subcycle, which therefore takes the value:

$$\mu_{2i} = \mu_2 \frac{x^b - x^a}{x^b - x^c} \tag{24}$$

In this way, inside each subcycle the dissipative term (through the slopes μ_{2i}) stores the memory of consecutive return points. Coupling a number of these elements in parallel transforms individual straight-line trajectories into global trajectories with smooth curvature. The model accurately reproduces the essential characteristics of hysteresis cycles; its main difficulty is the determination of the multiple parameters entering in the model.

This same idea of forcing the transformation trajectories to coincide at the return point, after and before having carried out a partial cycle, is at the basis of a phenomenological model proposed recently by Cesari et al. [37]; again, the difficulty of the method is to adapt the model to the experimental reality of a particular system.

Recently, another approach based on the Preisach model for ferromagnetic hysteresis [38,39] has been used to study pseudoelastic hysteresis cycles [32]. This time, the approach facilitates a direct connection with experimental data and, consequently, the possibility of quantitative prediction of any trajectory inside the two-phase region. On the contrary, it is not directly based on the thermoelastic balance. According to the model, transformation trajectories are given by the integral:

$$x(t) = \iint_{S^+(t)} \mu(\alpha, \beta) d\alpha d\beta \tag{25}$$

where $\mu(\alpha, \beta)$ is a population function, determined from a set of experimental first-order trajectories (for example, trajectories (-) that start at different x values of the main branch (+) and return to x=0). Memory of the return points, essential to the model, is retained by the particular shape of $S^+(t)$. A first application to pseudoelasticity in Cu-Zn-Al single crystals [32] has shown (a) a fine sensitivity of the trajectories to the precise values of experimental return points, and (b) an excellent reproducibility of L→H branches (branches (-)), much better than that of H→L branches, if a set of

first-order branches (-) was used to determine the population function of the model. This reflects a possibility that either the dissipation mechanisms or their relative importance differ from the forward to the reverse transition.

5.- Conclusions.

We have attempted to revise the thermodynamics of thermoelastic martensitic transitions, presenting a formalism that includes elastic and dissipative effects. We have given methods to evaluate the different energy contributions and presented experimental results showing their relative importance. Particular attention has been paid to the characteristic features of hysteresis in these systems, a subject of current interest; we have reviewed a number of phenomenological models recently proposed.

In our opinion, progress in this field should concentrate on (a) accurate computation of the elastic energy term in the thermoelastic balance, based on the existing knowledge of martensite microstructure, and its evolution along the transformation, and (b) determination of which specific mechanisms give rise to dissipation and how they lead to the observed macroscopic features of the transformation trajectories. We think that numerical simulations, in the line originally implemented by Khachaturyan [5], could be of great help to fulfill this objective.

Acknowledgements.

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