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Shape memory alloys: from high-resolution thermal analysis to predictive modelling and simulation

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Abstract

Using high-resolution automatised equipment (close to 0.001 N, 0.1 μm and 0.005 K), detailed observations can be made of the surfaces stress, strain and temperature (σ, ε, T) of Cu–Zn–Al shape memory alloys. The analysis of the experimental curves allows a model of the behaviour of the alloy to be formulated.

The model deals with a group of independent plates with a limited maximum growth and it separates the total friction into two parts: the nucleation of the domains of martensite or austenite (parent phase) and the friction of the movement of the interphases (the martensite growing–shrinking process). It is based on the existence of thermoelasticity/pseudo-elasticity. For local changes of composition and/or atomic order, the local temperature of transformation of the parent phase is assumed to be slightly different. The equation of Clausius–Clapeyron regulates the relationship between the external fields (temperature and stress). The results of the model with respect to changes in temperature and external stress, are consistent with the experimental measurements.

INTRODUCTION

There are two potential fields for the practical application of shape memory alloys (see, for instance, the general references 1–5). They can be used as systems with two different shapes in two-state systems in the same way as they are used in temperature control and conectics (for example, electric/electronic switches or links in computers), using the two forms of the material: the cold form, in the martensite phase, and the warm form, in the austenite or parent phase. In this case, the two shape memory effects would be used and an appropriate training process would be required.

The second possibility would exploit, in a continuous manner, the pseudo-elasticity/thermoelasticity phenomena. This would mean a predictive knowledge of the evolution of the alloys on the stress–strain–tempera-

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ture surface, which would allow their use in robotics. The characteristics of the material suggest other possibilities; for example, the existence of the hysteresis loop indicates that the material could be used to absorb vibrations.

The practical applications are limited by two essential conditions, one fundamental, the other economical. The first regards the problems associated with using a phase transition between metastable phases, and the second condition requires that the new device made with the alloy has to be more reliable and more cheap than the parts that it will replace. This is the reason why we only work with alloys based on Cu (as Cu–Zn–Al) which are much cheaper than those of Ti–Ni (memory steels are another lower price material).

The fundamental problems are on different levels. For the industrial preparation of the alloy, a well-determined composition has to be guaranteed. An uncertainty in tenths of a percentage of composition would alter the temperature in tens of degrees. In addition, the additives and thermal treatments necessary to obtain a thread or a plate of practical interest (in polycrystalline material) make the preparation of the alloy a delicate matter.

The problems that appear while analysing the behaviour of the polycrystalline material against time and cycling, are partially masked by the interactions of martensite with the surface of the grains. The use of single crystals allows some of the difficulties associated with the transformations and with the microscopic phenomena that take place during it to be more clearly visualized. The observations in controlled conditions allow the remarkable influences of the initial thermomechanical treatments to be established. Depending on the initial conditions, there are different concentrations of dislocations, of vacancies, of order domains, etc., that affect decisively the initial behaviour and its evolution [6–8]. A group of recent observations has established that the dynamic behaviour of the alloy is highly influenced by the initial concentration of dislocations [9,10] and, moreover, that the cycling process induces the creation of new dislocations (possibly because of the small change in associated volume and/or the interactions between variants).

The creation of dislocations is the problem that interferes with the use of the alloy. The classical training methods of the material create dislocation arrays. This creates a preferred direction and consequently induces the appearance of the variants of martensite that give the form that we are interested in. When we use the trained material, the cycling creates new dislocations that progressively destroy the initial training. To avoid this we use mixed systems that have springs of the alloy with memory, as well as classical steel springs [11,12]. The mixed system allows the alloy with memory to work only in the process of retransformation (martensite to austenite). Research into new training methods that would not change the

structure of the material so much is an open field for investigation. For example, the use of stabilised martensite has been suggested [13,14]. In this case the creation of a network of stress-induced small plates, stabilised by temperature, trains the material not transformed and produces the preferred direction of transformation. The possibilities of materials with precipitates present another field of research [15,16].

The fundamental analyses establish the existence of a secular evolution of the temperatures of transformation towards a final value that seems stable. Within an interval of about one year, it is possible to reach a stationary situation. For example in the practical applications of the control of temperature at room temperature, it has been necessary to use well-controlled alloys. A group of tests have allowed a series of thermomechanical treatments to be established that accelerate the evolution of the alloy and ensure the constancy of the transformation temperatures [12]. It seems that the interphase plays an important role in this process of acceleration towards a final steady state. In fact, different observations of fundamental character show that the coexistence of the two phases and, in particular, the movement of the interphases induces an important acceleration of the evolutions with time of the temperatures of transformation. That is to say, in the characteristics of the hysteresis cycle [8].

Once the alloy does not change with time [6,12], we can look for a predictive description of its behaviour based on a group of observations. To do this we have to establish a mathematical model that describes the behaviour on the surface of σ, ε and T. It is clear that practical applications need this type of formulation and, recently, more fundamental and/or more applied descriptions and patterns can be found in the literature (for less recent papers, see ref. 17 and related references). In general, the models are related to a thermodynamic representation of a free energy [18,19]. They presuppose that a stress-free transformation is achieved at constant temperature, or that if it implies the creation of new domains of martensite, it will be necessary to include the associated energies in the creation of new surfaces [19].

A preliminary analysis to describe the elementary stress-free processes of growing–shrinking associated with the changes in temperature, requires a high resolution; much higher than that used in classical metallurgical equipment (± 0.5 K) [20], of about ± 0.01 K or even less [6–10,21–26]. Commercial DTA or DSC equipment that are very versatile do not have sufficient resolution for a detailed description. Moreover, the study of partial cycles needs a high reproducibility of the extreme temperatures and temperature rates of the partial cycles. Even unconventional calorimetric systems, suitable for a detailed description of the phenomenology of the transformation, present substantial difficulties in the accurate measuring of the total exchanged energy. Furthermore, the usual observations frequently correspond to spontaneous transformations that are hard to interpret

through thermodynamics. In fact in the recent past, different thermodynamic descriptions [27–29] of the process of transformation–retransformation have been made that are essentially wrong. Some of the recent criticism can be found in refs. [16,30–32]. The fundamental problem seems to be associated with the uncertainties in measuring a process of low-level heat-power production (30–50 K), and where the mechanisms of the processes of transformation–retransformation have not yet been clarified [31,33,34].

To analyse accurately the process that takes place in the material it has been suggested that it would be convenient to observe, directly or indirectly, the phenomenology of the transformation. To achieve this, video systems have been incorporated into high-resolution thermal analysis (HRTA) equipment, or coupled measurement systems which can take a minimum of two independent measurements have been used [21,35]. The experimental observations establish that it is possible to study the behaviour of a single domain of martensite under the action of highly controlled, stress-free thermal cycling. These experimental conditions allow reproducible and "predictive" observations and have established that the processes of growing–shrinking are determined by the intrinsic thermoelasticity [7,10,36,37]. The friction associated with the movement of the interphases is very small and it is strongly influenced by the state of the surface. This means an essential change in the description of the process of growing–shrinking. Up to now it was considered to take place at constant temperature and the thermoelasticity was assumed to be associated with the interactions between the different variants and/or with the grain surface if a polycrystal is used.

The observations have suggested that in order to establish an effective modelling of the behaviour of the material, we need to have a clear phenomenology in the coordinates of stress, strain and temperature. In this paper we describe a system of automatised stress that allows the behaviour of samples of small section (≈ 0.5–1 mm^2) to be studied with an equivalent resolution in the three coordinates [16,34,38]. The group of experimental observations employed allows the relevant parameters of transformation–retransformation to be visualised: the friction associated with the movement of the interphases, the processes of nucleation in austenite and martensite, the intrinsic thermoelasticity and pseudo-elasticity, the limited growth of the domains of martensite, etc. The formulation of the experimental observations allows a model that is consistent with the behaviour of the alloy to be formulated.

EXPERIMENTAL SET-UP

A schematic outline of the experimental system is shown in Fig. 1. The system uses a PC-XT that controls the temperature plates (via GPIB) and

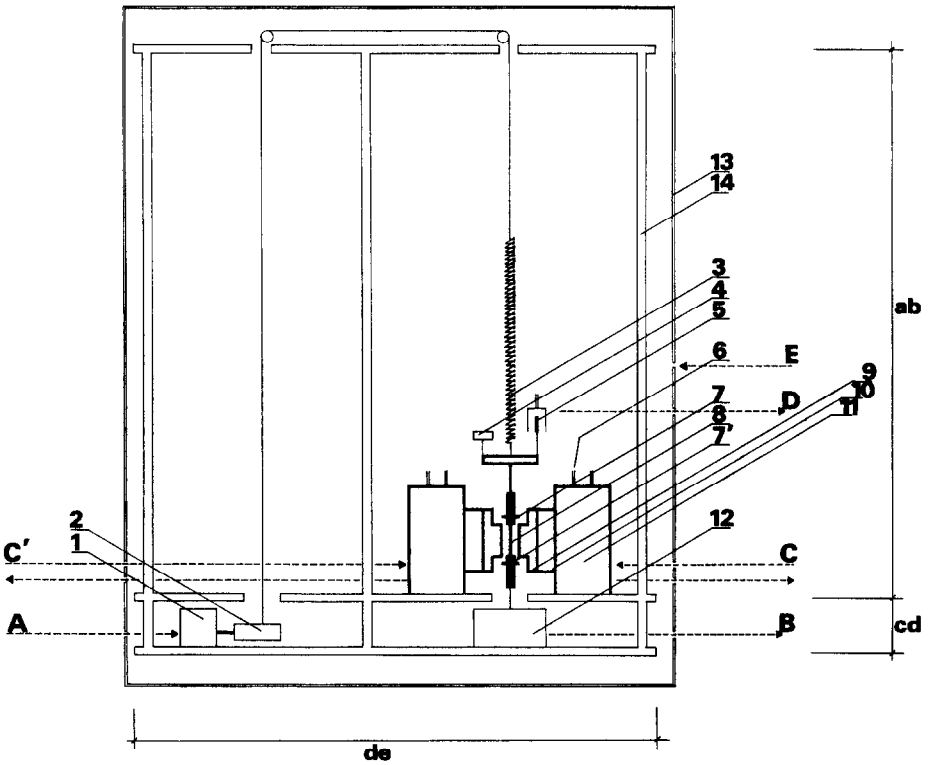

Fig. 1. Experimental set up (schematic diagram). Key: 1, stepper motor; 2, coil; 3, spring; 4, compensatory mass; 5, length measurement device; 6, link to the external thermostat; 7 and 7′, grips; 8, sample; 9, controlled temperature plate; 10, Peltier thermobattery; 11, reference temperature bath; 12, force measurement; 13, protecting chamber; 14, system structure in aluminium; A, motor control; B, force output signal; C and C′, temperature control by resistance measurement and Peltier effect against d.c. current; D, length measurement; E, isothermal chamber (temperature controlled by the external thermostat). Dimensions: ab, 850 mm; cd, 150 mm; de, 300 mm.

the advance of the motor (via RS-232) and takes the readings of force and displacement (GPIB). We have designed grips that are very movable and adaptable to the sample. They are very simple because the maximum force they have to apply is only 20 N. The rotation of the motor, which varies the length of the cable (steel covered with nylon, fishing type) changes the length of the steel spring. The design makes the hysteresis of the equipment irrelevant. The stiffness of the system can be observed on the experimental curves. The most important part of it is produced by the force sensor (300 μm under full load). For deformations of the alloy up to ≈ 1 mm, there are no important changes of the stress tensor σ_{ij} (see the torque perturbative effect in Fig. 2). The system operates in very slow conditions so that the energies interchanged in the processes of growing–shrinking do not affect the results of cycling.

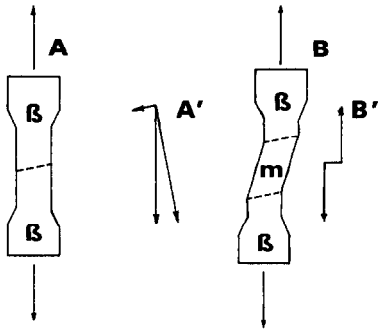

Fig. 2. Sample form and schematic forces diagram. Key: β and m represents the parent and the martensite phase; A, sample in parent phase with a habit plane trace; A', forces diagram over the habit plane; external, normal and shear are outlined; B, macroscopic strain in a single martensite domain (heat treatment TT1 type for a bigger plate); B', torque induced by the transformation strain.

A system of computerised displacement from Charly-Robot S.F. Weeq S.A. is used to apply the stress. It has a stepper motor (W07 3465) and a reducing system to increase the resolution (Oriental motor 2GK25K). It is controlled by a converter ID 3304 connected to the computer via the RS-232. In this way the rotation of the motor, which varies the length of the cable (steel covered with nylon, fishing type) produces changes in the length of the spring. In this work, the spring is 30 cm long and the recovering constant used is 2 N cm^{-1}. One step motor induces $\Delta f \approx 1.2$ mN.

For measuring the force and the displacement, accessories from Hottinger Baldwin Messtechnik (HBM) were used. In the first case we use a U1 cell of 20 N nominal charge (HBM) through an amplifier (MVD2405A) that gives an easy reading in kp and produces an analogue signal (full scale ± 1 V). The signal is digitised through a DMM HP 3478A and sent to a computer through a GPIB link. In the measuring of the lengthening, an inductive transducer W1E is used through the MC2A amplifier. It gives a signal of ± 5 V for displacements of ± 1 mm and it is digitised in the same way as the force.

The temperature is controlled through a double system of high-resolution thermal analysis based on the Peltier effect (see refs. 16, 25, 26, 33). The two plates are controlled and programmed independently with a resolution close to 0.003 K. This is why the temperatures are measured through Pt-100 resistances (resolution 0.001 ohm) and the Peltier thermobatteries are fed with d.c. current from a power supply (Premium SR-120) controlled by a D/A converter of 14 bits installed in the PC-XT. To reduce the variations in the external room temperature, the whole mechanism is placed inside thermal protection connected to an exterior thermostat (Lauda RMT-D6). Sometimes, independent observations by optical mi-

croscopy (Olympus BH2-UMA) and videorecording (Sony) were used to observe the local behaviour of the sample [33,38].

EXPERIMENTAL RESULTS

Single crystal samples of Cu–Zn–Al of electronic concentration 1.48 e/a (nominal $M_s \approx 288$ K) were used. The shape of the samples can be seen in Fig. 2 where the creation of a variant is shown schematically. To observe the nucleation we have used samples under air quenching (TT1) following 10 min at 850°C. We have observed the growth of many plates for an identical sample but after a water quenching (TT2). In each case the material was electrolytically polished after the thermal treatment, and the observations were made between one week and a few months after the thermal treatment. The temperatures of transformation associated with the TT1 are almost 10 K higher that those of the TT2 which are associated with the initial atomic order induced by quenching type [8,10].

The effect of the nucleation and the difference between nucleation and friction linked only to the displacement of the interphases can be seen in Fig. 3 (for only one plate TT1). Many plates (for an identical sample) take part in the group of cycles shown in Fig. 4, here after a water quenching (TT2). In Fig. 5, the phenomenology associated with the partial cycles shown in Fig. 4 can be seen in more detail. Figure 6 deals with the changes in position of the representation force against sample length when the temperature changes (see, in Fig. 7, the representation of the force against time and temperature). This group of observations shows that in order to describe the phenomenology of the transformation, it is necessary to consider that:

(1) The observations of the spontaneous transformations and of those that take place under the action of a low external stress establishes that the

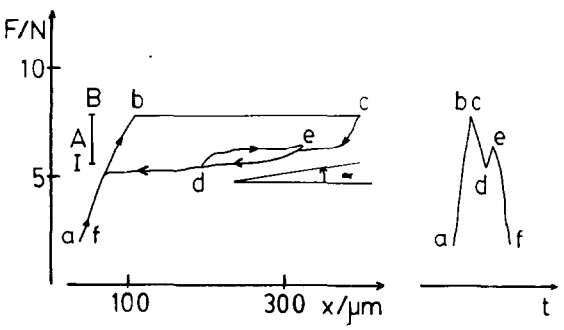

Fig. 3. Left: force (F) against increased length (x) near 295.2 K. Heat treatment: air-quenching or TT1; slope in ab is the system stiffness (≈ 15 μm N^{-1}); the slope in ed ($\approx \tan \alpha$) represents the intrinsic pseudo-elastic effect; ded, frictional partial cycle produced by the interface movement; A, frictional force; B, nucleation (parent to martensite). Right: force versus time; a, b, c, d, e, f, homologous points.

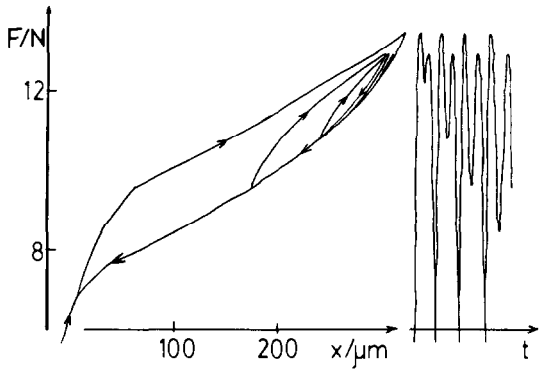

Fig. 4. Left: force (F) versus increased length (x) near 289.5 K. Heat treatment: water-quenching or TT2. Right: Force versus time.

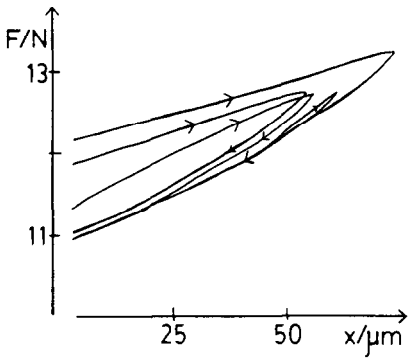

Fig. 5. Force against displacement (the upper part of Fig. 4).

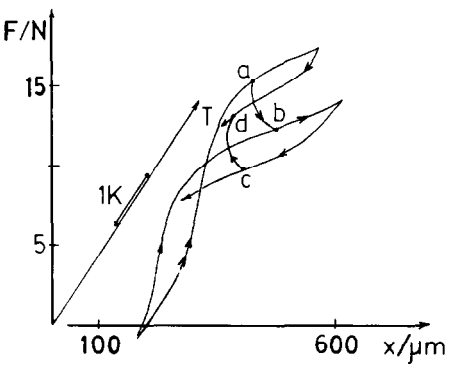

Fig. 6. Force (F) versus increased length (x) between 291.3 and 292.3 K. Heat treatment: water-quenching or TT2; ab and cd changes at constant force from upper to lower temperatures and vice-versa. Schematic representation of force and temperature changes are presented in Fig. 7.

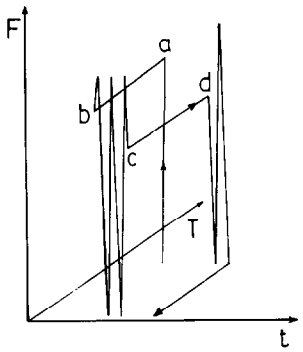

Fig. 7. Schematic representation of force versus time and temperature used in Figs. 6 and 10; ab and cd changes (at constant force) from upper to lower temperatures and vice versa.

creation of plates does not take place simultaneously on all of the material. The existence of small differences of composition probably causes the temperature of transformation T_0 to be slightly different in different parts of the same monocrystal.

(2) For the nucleation, to produce martensite, a supplementary undercooling is necessary or an equivalent critical stress of about 1–2 K. The creation of a plate of martensite like the one in the outline in Fig. 2 is a complex process. First a needle (microplate) has to be created and by its growth it has to completely cross the sample. The mechanisms of growth of the microplate and the plate are different. In the first it is necessary to overcome the internal tensions between the austenite phase and the needle. The microplate growth induces a skew dislocation displacement and needs supplementary work. At the end of the growth of the microplate the system is unstable and the plate of martensite grows spontaneously. The processes of growing–shrinking of the microplates extend greatly the domain of transformation/retransformation of the plates. In fact, the material is in the process of transformation in the presence of microplates, but the quantity of martensite is still negligible and the total strain is very low. In the process of retransformation new plates of austenite can nucleate. This can readily be visualised in samples with γ precipitates and it can be seen that the nucleation with austenite needs lower overheatings (≤ 0.5 K).

(3) The friction associated with the movement of the interphases is very small. The lowest values observed are about 0.1 K and they change depending on the plate under analysis. This value is highly affected by the state of the surface and by the secular evolution of the material. For observations made hours or days after the thermal treatment, an important part of the friction is associated with the processes of stabilisation of the parent phase and the recuperation of the martensite.

(4) The displacement of the interphases is controlled by the intrinsic thermoelasticity/pseudo-elasticity linked to the concentration of disloca-

tions. For materials under thermal treatment TT2, the initial concentration is high. To make the martensite grow it is necessary to have a significant (and increasing) thermodynamic force and this assists the creation of new plates. The higher value of plate length in TT1 approaches ≈ 1 mm. In TT2, the length is ≤ 100 μm.

MODELLING AND SIMULATION

The behaviour of the material establishes the basic characteristics of the model [39]. The essential mechanisms that determine the state of the transformation are the temperature T_o of each domain of the crystal, the nucleations and the concentration dislocations. In the model we assume that the material, under an external force, would not produce more than one variant and we divide the material into N domains ($i = 1, 2, \ldots, N$) that correspond to the N plates that can be formed. For each domain we assume there is a temperature of transformation T_o^i or, in the same way, a critical stress (force f_c^i) to transform. For the friction we take the same value for all the plates multiplied by a random value between 0 and 1.

To simulate the complex process of creation and disappearance of a plate we assume that nucleation includes all the processes of creation of the microplate until the plate grows. The disappearance takes place in two stages. If the reduction of the external force (or the heating) is not higher than a predetermined value, a microplate can remain which can grow without nucleating.

The program is organised through a series of indexes that establish the state of each of the plates and of the variations of the external fields (force and temperature). For each new value of the external field, the initial state of each plate is analysed, its new state determined and the indexes updated. The addition of each one of the lengthenings gives us the total lengthening (strain) of the material.

Figure 8 is an equivalent simulation to the experimental curve of Fig. 3. It is the simulation of the behaviour of only one plate (force against strain) and of the associated external force. We can clearly see the effect of the nucleation, the friction and the pseudo-elasticity. Figure 9 is a cycling equivalent to the one done in the tests and presented in Figs. 4 and 5. The agreement between the partial experimental cycles and the simulated ones is quite satisfactory. Figure 10 simulates a cycling associated with the changes in temperature equivalent to the ones obtained through tests and shown in Fig. 6. The effect of the temperature is introduced with the help of the Clausius–Clapeyron equation ($\mathrm{d}f/\mathrm{d}T \approx$ constant). A connection between the effective value of the field of external forces (f and T) and the value of f_c used in the calculation is established through the equation

$$f_c = f_c^* + (\mathrm{d}f/\mathrm{d}T)(T - T_{amb})$$

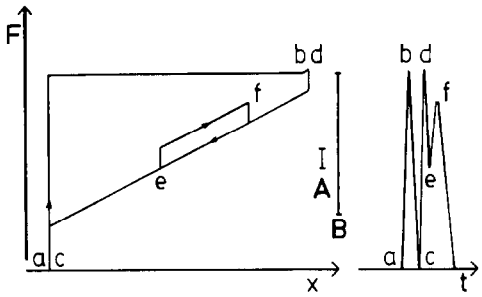

Fig. 8. Computer simulation. Growing/shrinking of a single martensite plate (see experimental measurement in Fig. 3); a, b, c, d, e, f, homologous points. Left: force versus displacement, fluctuations are connected to the plate nucleation processes (parent to martensite and martensite to parent phase); A, friction for the interface movement; B, force to overcome the nucleation effect (parent to martensite phase). Right: force against time.

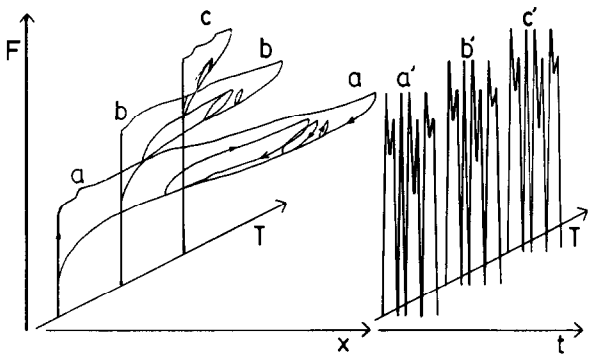

Fig. 9. Computer simulation of a "water-quenched material". Growing/shrinking of different martensite plates at three temperatures (see the experimental measurement presented in Figs. 4 and 5); a and a′, b and b′, and c and c′ are the homologous curves. Left: force against displacement and temperature. Right: force against time and temperature.

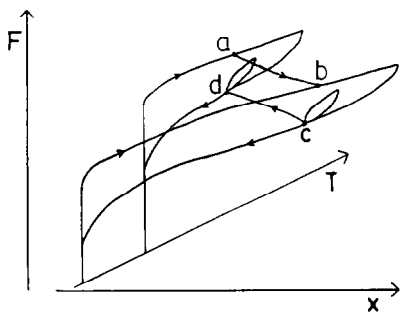

Fig. 10. Force versus displacement and temperature. Computer simulation of a "water-quenched material", ab and cd changes (at constant force) from upper to lower temperatures and vice versa. Simulation "equivalent" to the experimental measurement presented in Fig. 6.

where f_c^* is the critical force if the external temperature is room temperature ($T = T_{amb}$).

The agreement of the model with the experimental observations suggests a possible identification with a physical image that would allow the automatic establishment of a representative model of a material. The preliminary analyses (made in a way parallel to that of the method outlined in ref. 31) seem to indicate that this is possible.

CONCLUSIONS

The construction of a computerised, adapted system of stress–strain–temperature with resolution close to 0.005 K, 0.1 μm and 0.001 N allows the stress–strain–temperature surface of Cu–Zn–Al shape memory alloys in single crystals to be studied and the more relevant phenomena of the processes of transformation–retransformation to be visualised.

The building of a model of growth of independent plates that includes the nucleation of martensite and parent phase, the intrinsic thermoelasticity–pseudo-elasticity and the friction associated with the movement of the interphases has enabled a satisfactory agreement between the results of the model and the experimental observations to be obtained. The model simulates the behaviour of the alloy in the global and partial cycles and it suggests that automatically established routines of identification of the behaviour of the material can be made.

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