

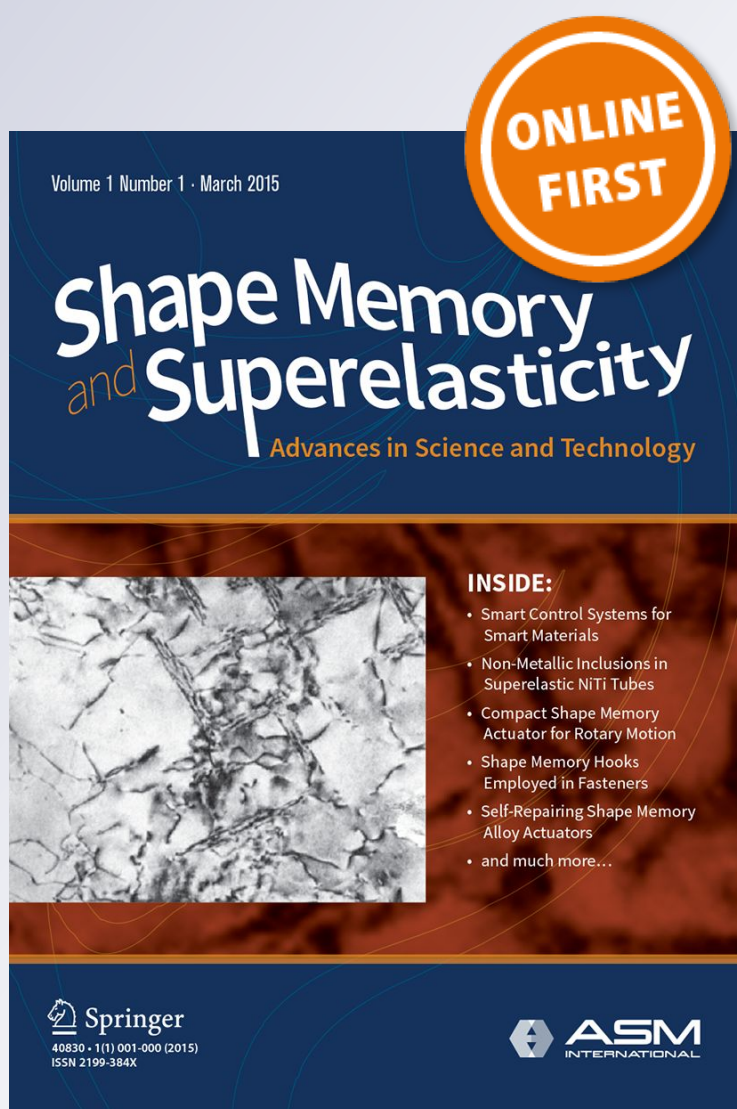
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Shape Memory and Superelasticity
Advances in Science and Technology

ISSN 2199-384X

Shap. Mem. Superelasticity
DOI 10.1007/s40830-018-0155-2



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The Relevant Role of Dislocations in the Martensitic Transformations in Cu–Al–Ni Single Crystals

 R. Gastien¹ · M. Sade^{2,3,4} · F. C. Lovey^{2,4}

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Abstract The interaction between dislocations and martensitic transformations in Cu–Al–Ni alloys is shortly reviewed. Results from many researchers are critically analyzed towards a clear interpretation of the relevant role played by dislocations on the properties of shape memory alloys in Cu-based alloys. Both thermally and stress-induced transformations are considered and focus is paid on two types of transitions, the $\beta \rightarrow \beta'$ and the formation of a mixture of martensites: $\beta \rightarrow \beta' + \gamma'$. After cycling in the range where both martensites are formed, the twinned γ' phase is inhibited and cycling evolves into the formation of only β' . A model which considers the difference in energy of each γ' twin variant due to the introduced dislocations quantitatively explains the inhibition of γ' in both thermally and stress-induced cycling. The type of dislocations which are mainly introduced, mixed with Burgers vector belonging to the basal plane of the β' martensite, enables also to explain the unmodified mechanical behavior during $\beta \rightarrow \beta'$ cycling. The reported behavior shows interesting advantages of Cu–Al–Ni single crystals if mechanical

properties are comparatively considered with those in other Cu-based alloys.

Keywords Cu–Al–Ni · Shape memory alloys · Cycling · Dislocations · Pseudoelasticity

Introduction

Shape memory materials are useful as sensors and actuators as they offer a precise temperature and displacement range response together with a quick and reversible reaction at external stimulus. Peculiar martensitic transformations are the responsible of the shape memory and pseudoelastic behavior of these materials [1, 2]. An optimization of mechanical and thermal properties of these alloys is of fundamental importance to obtain better performances. In this way, the correspondence between microstructure and thermo-mechanical response is a relevant subject to analyze in order to predict the material behavior. Cu–Al–Ni alloys are particularly interesting among other Cu-based shape memory alloys because they are candidates for moderate temperature applications, mainly due to their good resistance against stabilization mechanisms above room temperature.

In Cu–Al–Ni, two martensitic transformations are responsible for the shape memory and pseudoelastic properties; $\beta \leftrightarrow \beta'$ and $\beta \leftrightarrow \gamma'$, where β' and twinned γ' correspond to the usually named 18R and 2H martensites, respectively [1]. The composition range at which each of these transformations takes place has been reported in [3, 4]. Both transitions can be thermally or stress induced, being M_S , M_F , A_S , and A_F the critical transformation temperatures for the start and end of the transformation and retransformation, respectively.

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The martensitic transformations in Cu–Al–Ni alloys are characterized by a small volume change, which is the main reason for their thermoelasticity. One of the main properties of thermoelastic transitions is that some kind of energy is stored during transformation and released during retransformation. At least two mechanisms are envisaged to explain this behavior. Namely, the creation of stacking faults and interfaces due to the interaction with existing dislocations [5], and some elastic energy accumulated in regions where variants from different accommodations groups impinge each other. Larger volume changes characterize non-thermoelastic transitions, which usually require microplasticity to accommodate deformations which are introduced for the transformation to take place. In the case of Cu-based alloys, although the volume change is small, it has been shown that dislocations are introduced in the alloys if thermal or stress-induced cycling is performed. These dislocations might affect the properties of the material, modifying transformation temperatures, critical stresses, and hysteresis.

In this work, the authors shortly review the research made on the interaction between martensitic transformations and defects in Cu–Al–Ni single crystals. The presence of defects, mainly dislocations, has interesting and specific consequences on the stress-induced martensitic transitions, altering for example the relative stability of the phases. In this respect, it was found that the ratio of the β' to γ' martensites changes during $\beta \leftrightarrow \beta' + \gamma'$ pseudoelastic cycling, until only the $\beta \leftrightarrow \beta'$ martensite takes place. Microstructural observations and a microscopic model analyzing this interaction are remarked in this report.

Experimental Procedures and Results

The experiments consisted in mechanical tensile cycling of single crystals specimens of various chemical compositions around Cu–14Al–4Ni (wt%). In all cases, the samples were previously thermally treated 1 h at 1173 K and quenched in water at 273 K. Tensile cycling was performed at well-controlled temperatures which allowed selecting the involved martensitic transformation. In addition, defects left in the material after cycling were analyzed and characterized by transmission electron microscopy (TEM). See Ref. [6] for details.

It has been shown that for Cu–Al–Ni single crystals of composition near Cu–14Al–4Ni (wt%) different behaviors are found if a single crystal of the β phase is tensile stressed [7]. At temperatures above M_S but sufficiently close to this temperature, the 2H(γ') martensite is formed. Unloading the material usually leads to have martensite at zero load due to the large hysteresis of this transformation. Heating the material enables the retransformation to the β phase,

which corresponds to a shape memory behavior instead of a pseudoelastic one. At higher temperatures, tensile loading the β phase leads to the nucleation and growth of a single martensite crystal with 18R structure (β'), which retransforms to the β structure if the material is unloaded. An interesting fact arises if tests are performed at an intermediate temperature range, which is rather small, where a mixture of both martensitic structures is formed. In this case, pseudoelasticity is present, showing a hysteresis larger than the present one if only 18R martensite is formed.

In the case of the $\beta \leftrightarrow \beta'$ transition, no evolution of the stress–strain curves can be observed up to nearly 3000 cycles in specimens oriented close to the $[100]_\beta$ direction [6]. When stress-induced martensitic transformations are induced repeatedly in a shape memory alloy, certain defects are generated in the crystal structure, being dislocations the most important ones due to their irreversible character. The analysis by TEM showed that the dislocations are partially arranged in bands [6], being the dislocation of the mixed type with Burgers vector of the type $\langle 100 \rangle_\beta$ and line direction preferentially along $\langle 111 \rangle_\beta$ directions. One important characteristic of the majority of these dislocations is that the gliding plane is of the type $(0\bar{1}1)_\beta$. Such plane transforms into the basal plane of the induced β' martensite. These defects interact with the martensitic transformation each time that it is induced, generating changes in the thermal and mechanical response of the material. However, the effect might be different according to the type of transformation taking place.

The density of the introduced dislocations is not as large as the present in Cu–Zn–Al cycled single crystals [8, 9] for similar number of cycles and test temperatures. However, the type of dislocations is the same as reported for Cu–Zn–Al tensile cycled samples. A main point to be emphasized here is that dislocations introduced during $\beta \rightarrow \beta'$ pseudoelastic cycling do not alter the relevant parameters which characterize this transformation. Additionally, no phenomena associated to diffusion either in austenite or in martensite are visible, enhancing the potential uses of this alloy.

A different and remarkable situation arises if pseudoelastic cycling is performed in the temperature range where a mixture of martensites is formed. Tensile stress-induced $\beta \leftrightarrow \beta' + \gamma'$ cycling was performed on Cu–Al–Ni single crystals samples of different crystal orientations. An important change of the σ – ϵ (stress–strain) curves was observed as the cycling progresses. Namely, after 400 cycles the hysteresis reduces drastically showing only the $\beta \leftrightarrow \beta'$ transformation (see Fig. 1).

Those results show an inhibition of the 2H structure as the pseudoelastic cycling evolves. This effect was also observed in $\beta \leftrightarrow \beta' + \gamma'$ thermal cycling [10]. A deep

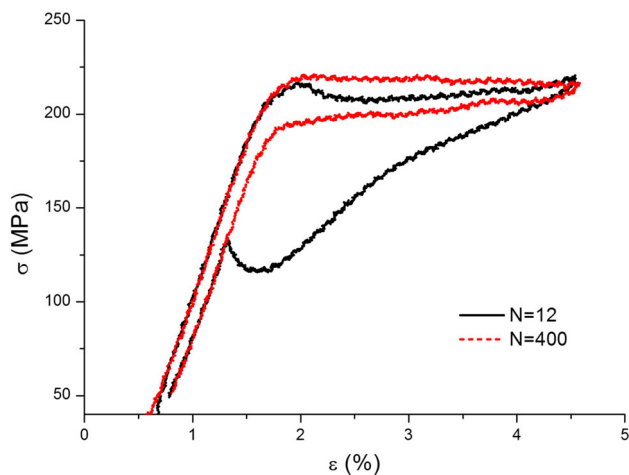


Fig. 1 Mechanical evolution after pseudoelastic cycling under tensile mode in the temperature range at which a mixture of martensites is stress induced. Cycles 12 and 400 are shown. Composition of the single crystal: Cu–14.3Al–4.1Ni (wt%), orientation of the tensile axis: $[436]_{\beta}$, test temperature = 249 K

analysis of the interaction between the martensitic transformation and defects generated during this kind of cycling was made by the authors and was the clue to understand this particular behavior [6, 10–12].

An experiment for measuring the driving force needed to induce the γ' martensite after being inhibited in tensile $\beta \leftrightarrow \beta' + \gamma'$ cycling was described in [10]. In that experiment, a $\beta \leftrightarrow \beta' + \gamma'$ cycle was performed at a temperature named T_{exp} . After tensile cycling at that temperature, the pseudoelastic response evolves to $\beta \leftrightarrow \beta'$. Then the temperature was lowered until a $\beta \leftrightarrow \beta' + \gamma'$ cycle was recovered in a further cycle. The temperature at which it happened was called T_{rec} . The difference $T_{\text{exp}} - T_{\text{rec}} = \Delta T_{\text{in}}$ was considered as the γ' inhibition temperature difference and an average value of 10 K was obtained. An estimation of the inhibition energy was made using this value and entropy determinations [7]. A value for this energy of around 15 J/mol was obtained.

Observations in a transmission electron microscope of the bulk of the samples after $\beta \leftrightarrow \beta' + \gamma'$ cycling were performed by the present authors [6, 10]. Dislocations of a mixed character with Burgers vector direction corresponding to a $\langle 100 \rangle_{\beta}$ direction and line direction of the $\langle 111 \rangle_{\beta}$ type forming bands were observed. An example of the observed dislocations is shown in Fig. 2.

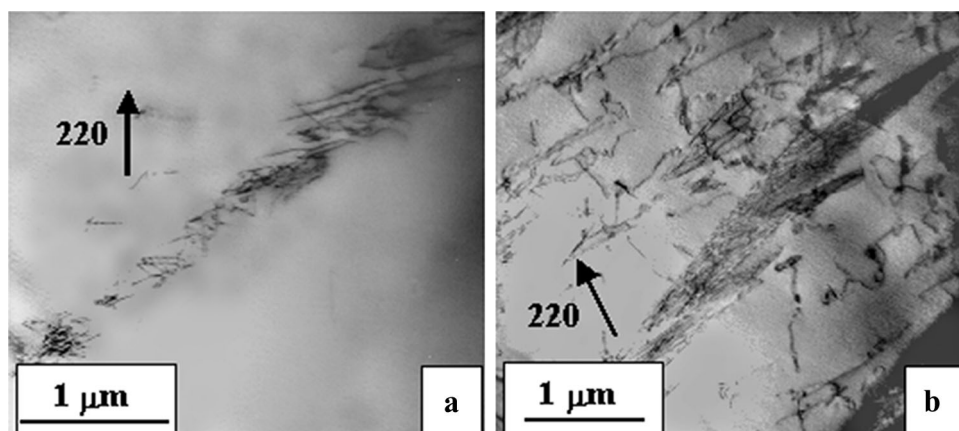
The dislocation density increases as cycling progresses. According to Rodriguez et al. [13] the difference of the elastic energy of the dislocations when embedded in β phase or in the β' or γ' martensites would be very small. Therefore, the inhibition of the γ' phase after pseudoelastic cycling is more likely related to the twinned character of the γ' martensite and the different interaction of each twin variant with the dislocations bands.

The growth of a martensite plate requires the formation of a macroscopically undistorted habit plane. In the γ' martensite, a twinned microstructure is necessary, to provide a lattice invariant shear, with a specific twin width ratio (about 2:1). Our model [12] is related to the localized interaction between a dislocation array and the twinned γ' structure. Dislocations with Burgers vector $\langle 100 \rangle_{\beta}$ and line direction $\langle 111 \rangle_{\beta}$ (accordingly to the microscopic observations) in an isotropic β matrix are considered.

Two main mechanisms were considered for these interactions. One of them comes from the stacking faults created every time the Burgers vector of a dislocation in the β phase lies out of the basal plane of the martensite [6]. The other contribution, taken into account, originates in the interaction between the martensitic stress-free transformation strains and the stress field created by the dislocation arrays.

It has been shown in Ref. [12] that the interaction is different for each twin-related variant in the γ' martensite. This situation generates an energetic unbalance that favors the formation of one twin variant with respect to the other. The average value of the calculated energy difference is

Fig. 2 TEM images of dislocation bands after 2400 cycles. **a, b** Correspond to different areas of the foil. Reproduced with permission from [6] Elsevier



$E_{inh}^T \approx 12$ J/mol. If one of the twin variants becomes energetically more favorable the formation of the right twin ratio will demand an extra energy. Since the entropy change of the material due to the dislocations and stacking faults is very small, the E_{inh}^T can be regarded as an increase of the driving force to induce the γ' transformation. This driving force can be provided either by applying stresses or cooling. The calculated E_{inh}^T value is in agreement with the one obtained using experimental data in [10], described above. It is remarkable that the results of the inhibition energy obtained by this model change slightly with the number of dislocations that conform an array. This feature highlights the localized character of this interaction.

On the other hand, the β' martensite has a very weak interaction with the dislocations. Therefore, a larger driving force to attempt the formation of the γ' inhibits its formation and would first induce the β' martensite. Such phenomenon can be analyzed by the use of σ - T phase transformations diagrams. Once the γ' is inhibited at a certain test temperature, it is necessary to decrease the temperature to recover the original $\beta \leftrightarrow \beta' + \gamma'$ cycle, which means an increase of the driving force to induce γ' again. This can be thought as a relative displacement of the $\beta \leftrightarrow \gamma'$ transformation line towards lower temperatures, as indicated in Fig. 3. The $\beta \leftrightarrow \beta' + \gamma'$ cycle obtained at T_{exp} before the γ' inhibition was recovered at T_{rec} , and $T_{exp} - T_{rec} = \Delta T_{in}$ is the quantity the $\beta \leftrightarrow \gamma'$ line moves towards lower temperatures. In fact the equilibrium $\beta \leftrightarrow \gamma'$ line may also move (locally) somewhat to the left due to the creation of stacking faults. This effect, however, would not provide a contribution larger than about 2 J/mol and is not indicated in Fig. 3.

Concerning the markedly stability of the $\beta \leftrightarrow \beta'$ transformation on cycling, the following explanation was

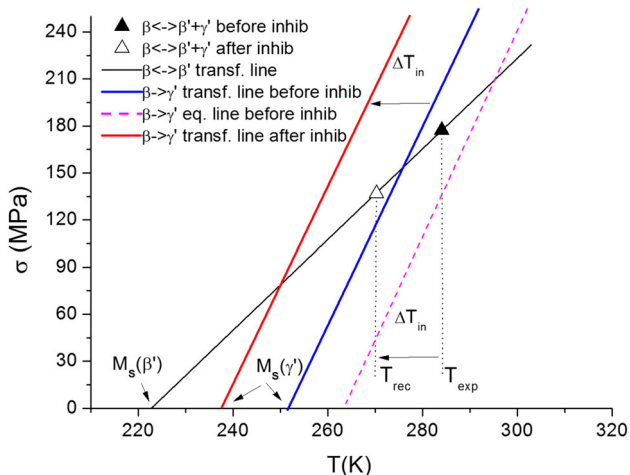


Fig. 3 Stress–Temperature (σ - T) diagram for a Cu–14.3Al–4.1Ni (wt%) single crystal. Tensile mode was used. See text for details

developed. The dislocations which are introduced during cycling are mainly of mixed character and their Burgers vector of the type $\langle 100 \rangle_{\beta}$. The Burgers vector of many dislocations belongs to the basal plane of the induced martensite, and derives from the $\langle 0\bar{1}1 \rangle_{\beta}$ plane. On one hand, it has been shown that the difference in elastic energy between having these dislocations either in β or in 18R is negligible [5, 13]. On the other hand, as the Burgers vectors of these dislocations are translation symmetry vectors in both lattices, no faults are dragged by the growing martensite for each absorbed dislocation. Conversely, for those dislocations having the Burgers vectors out of the basal plane a fault is dragged by the growing martensite giving place to the intrinsic thermoelasticity (i.e., a gradual decrease of temperature is needed to force a single plate to grow). However, since the nucleation energy of the 18R martensite is very small many parallel plates are created to offset the intrinsic thermoelasticity [14]. Hence, no significant changes either in the critical stresses or in the hysteresis of the transformation are expected.

Discussion

Martensitic transformations belong to the large group of solid–solid phase transformations, and nicely show the significant role played by the relative phase stabilities on the material properties. In this case involved structures, either the austenite or martensite, are metastable, and their existence is based on the extremely slow kinetics required to have the equilibrium phases at temperatures close to room temperature. Relative stability can be changed by the variation of thermodynamic coordinates like temperature or applied mechanical stress. Lowering the temperature or increasing the stress increases the relative stability of the martensite in comparison with the corresponding one to the austenitic structure. An additional interesting point emerges when relative stabilities among different martensitic structures also play a role. In fact, several martensitic structures have been reported in Cu-based alloys and in most of the cases composition plays the main role in the selection of the martensite to be formed. It has been reported in the most studied Cu-based systems that the relative phase stabilities among different martensites can be altered by applying stresses. A nice example of this is the formation of the 6R martensite which can be found after applying tensile stresses to the stress-induced variant of 18R in Cu-based single crystals with orientations close to $\langle 100 \rangle_{\beta}$ [15, 16]. On the other hand, thermally or stress-induced 18R and 2H are usually formed in Cu-based alloys. However, specific thermodynamic coordinates are required to have the nucleation and growth of both martensites

taking place simultaneously. This phenomenon is scientifically attractive but also has interesting consequences on the functional properties, particularly due to the significant difference in the involved hysteresis and in the different variations of critical stresses with temperatures (Clausius–Clapeyron relationships).

Different authors have reported material behaviors in Cu–Al–Ni alloys which can be explained by the nucleation or growth of both martensites, i.e., 2H and 18R. Concerning thermally induced transitions, the martensitic transformation from β to a mixture of martensitic structures ($\beta' + \gamma'$) has been reported in several references [17–19]. Seguí et al. [18] and Recarte et al. [19] show that, when the mixture $\beta' + \gamma'$ is thermally induced from β , some microplasticity is present due to an incomplete self-accommodation of both martensites, and a stabilization of the β' martensite with respect to the γ' martensite is favored during the reverse transformation. As the thermal cycling progresses, this plastic deformation would enhance the formation of β' , as it forms autoacomodant groups of variants easier than the γ' , and the internal stresses are better accommodated. Consequently, the amount of γ' martensite might decrease, as was observed in the thermal cycling presented in [10].

On the other hand, it is well known that an anisotropy on the response of stress-induced transformations in tension/compression is present if Cu–Al–Ni single crystals are considered, topic studied in detail by Novak et al. [20]. Ibarra et al. [21] analyzed the evolution of stress–strain curves after superelastic compression cycling in Cu–Al–Ni single crystals. In one of the compositions used by those authors, an inhibition of the 2H martensite is also reported. The authors noticed that cycling modifies the microstructure of the sample and they categorized two kinds of dislocations: type A dislocations that are responsible of increasing elastic and frictional energies and type B dislocations created by plastic deformation of the 2H martensite, which favor the presence of thermal martensite, shifting up the transformation temperatures and decreasing thermal hysteresis.

No quantitate analysis had been performed until the present authors proposed the model which explains the inhibition of γ' formation by the asymmetric effect of dislocations on the energy of each one of the twinned variants which are formed in order to get an invariant plane. The model enables to understand both inhibition effects, i.e., the observed one during thermal cycles and the present effect after pseudoelastic cycling. The reason is quite clear since dislocations which are left in the material after both types of cycles are of the same type [10, 22–26].

Finally, it is interesting to analyze the main differences between the pseudoelastic cycling of Cu–Zn–Al single crystals and the commented results obtained with Cu–Al–

Ni ones. In Cu–Zn–Al crystals, two different behaviors can be briefly mentioned: (a) a recoverable one mainly present at temperatures above room temperature where a dynamic inhomogeneous stabilization takes place; transformation critical stresses decrease during cycling, while a recovery is observed after stopping the cycling test and (b) mainly irreversible phenomena obtained after cycling at lower temperatures, where a noticeable increase in hysteresis and in transformation critical stresses are present. Details concerning point (a) and (b) can be found elsewhere and will not be deeply commented here [27]. However, those results contribute to consider some benefits concerning possible uses of Cu–Al–Ni single crystals. In these ones, tensile cycling through the β –18R martensitic transformation does not show noticeable changes in the stress–strain curves, up to the detected fracture. It is noticed here that the absence of a dynamic stabilization is a clear advantage of the Cu–Al–Ni system if compared with Cu–Zn–Al or even the behavior reported for Cu–Al–Be crystals [28]. In this last one, stabilization of martensite also plays a significant role during pseudoelastic cycling at test temperatures higher than room temperature leading to a strong and inhomogeneous decrease of transformation stresses. If we focus the attention on pseudoelastic cycling of Cu–Zn–Al single crystals at low temperatures, relevant microstructural changes are introduced in the material, where complex dislocation tangles with retained martensite inside them, behave as three dimensional obstacles for the martensitic transformation leading to an increase in transformation stresses and hysteresis. A harder Cu–Al–Ni matrix, where a smaller dislocation density is formed after $\beta \leftrightarrow \beta'$ cycles, shows stable mechanical properties when a single martensite is formed.

Conclusions

- Pseudoelastic cycling of Cu–Al–Ni single crystals has been shortly reviewed.
- No significant changes are observed if β – β' pseudoelastic cycles are performed
- γ' martensite is inhibited after $\beta \leftrightarrow \beta' + \gamma'$ cycling, either thermally or mechanically performed
- A model which considers the different effect of dislocations on the energy associated to each one of the twinned variants of the γ' explains the reported inhibition both qualitatively and quantitatively.

Acknowledgements It is a pleasure to contribute with this manuscript to a special issue which honors the extended and deep contribution of Professor Jan van Humbeeck to the Martensitic Transformations and Shape Memory research field. Scientific

discussions along many years and a nice friendship are truly acknowledged.

Funding Support from Universidad Nacional de Cuyo, Project 06/C516 is acknowledged.

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