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**Mechanická stabilizace v monokrystalech slitin
s tvarovou pamětí**

**Mechanical Stabilization in Single Crystals
of Shape Memory Alloys**

Summary:

Thermoelastic martensitic transitions in shape memory alloys are strongly affected by the ferroelastic domain structure. This effect is called mechanical stabilization of martensite and is macroscopically observable as a significant shift of the reverse transition temperature upwards when the material is pseudoplastically strained. In this habilitation lecture, the effect of the mechanical stabilization is studied on the level of single crystals for the prototypical copper-based shape memory alloy Cu-Al-Ni. It is shown, that the mechanical stabilization leads to formation of highly dissipative interfacial microstructures, and that the propagation of these microstructures is the main mechanism providing the reverse transition. The morphology and energetic stability of these microstructures is discussed both in the macro-scale and the micro-scale.

Souhrn:

Termoelastické martenzitické transformace ve slitinách s tvarovou pamětí jsou silně ovlivněny feroelastickou doménovou strukturou. Tento efekt se nazývá mechanická stabilizace martenzitu a makroskopicky ho lze pozorovat jako výrazný posuv transformačních teplot pro zpětnou transformaci v případech, kdy je materiál pseudoplasticky deformován. V této habilitační přednášce je efekt mechanické stabilizace zkoumán na úrovni monokrystalů prototypické slitiny s tvarovou pamětí na bázi mědi, Cu-Al-Ni. Je ukázáno, že mechanická stabilizace vede ke vzniku vysoce disipativních rozhraňových mikrostruktur a že pohyb těchto mikrostruktur je základním mechanismem zpětné transformace. Morfologie a energetická stabilita těchto mikrostruktur je diskutována jak na mikro-škále, tak na mikro-škále.

Klíčová slova:

Termoelastická martenzitická transformace; slitiny s tvarovou pamětí; mechanická stabilizace martenzitu; rozhranové mikrostruktury.

Keywords:

Thermoelastic martensitic transition; shape memory alloys; mechanical stabilization of martensite; interfacial microstructures.

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1 Introduction

Shape memory alloys (SMAs) are smart functional materials able to undergo a fully reversible solid-to-solid phase transition called thermoelastic martensitic transition [1, 2, 3]. This transition lies behind all unique macroscale behaviors of SMAs, such as the shape memory effect [4, 5], superelasticity, rubber-like hyperelastic behavior [6, 7, 8], or magnetically induced reorientation [10, 11]. Among the copper-based shape memory alloys, the Cu-Al-Ni system [12, 13, 14, 15] has been by far the most studied one during the past four decades (see e.g. [16] for a review). There are several reasons why particularly this alloy has been so extensively studied both on the theoretical level and by experimental observations: both the cubic high-temperature phase (*bcc* austenite) and the tetragonal low-temperature phase ($2H \gamma'_1$ –martensite) of this alloy have simple crystal structures, the transformation strains are large (up to 8.5 %, [13]), the transitions are fully reversible and are not accompanied by any significant plastic flow, the twinning stress can be as low as 0.7 MPa [17], and the transition temperatures can be set close to the room temperature. In addition, the surface energy of the twinning planes in this alloys is relatively high [18], and so the martensitic laminates and other regular microstructures forming in single crystals of this alloy appear at the length-scales of several tens of micrometers or even larger, which enables easy observations of these microstructures by optical microscopy.

These properties predetermine the Cu-Al-Ni alloy to be used as an illustrative example of the SMAs [5, 16, 19]. In this sense, the optical micrographs of habit planes, macrotwins or more complex microstructures in this alloy have been used several times to confirm the theoretical predictions of the mathematical theory of martensitic microstructures [20, 21], which explains the formation of the observed microstructures by means of energy minimization and kinematic compatibility.

One of the most interesting effects observed in the single crystals of the Cu-Al-Ni alloy is the *mechanical stabilization of martensite* [22, 23, 24], i.e. the strong dependence of the martensite \rightarrow austenite transition temperature on the initial microstructure of martensite. This effect occurs whenever the initial microstructure cannot directly form any compatible interface with the austenite phase, and thus, the nucleation of austenite in the crystal requires local reorientation of martensite in order to form some microstructural object providing kinematic compatibility between the initial microstructure and the austenitic nucleus. This object is called the *interfacial microstructure* [25, 26], and propagates through the material with the growth of the austenitic nucleus until the transition is completed. The energy required for the formation of the interfacial microstructure increases the nucleation barrier for

austenite, and so the transition is shifted to higher temperatures.

2 Theoretical background for the mechanical stabilization effect

The Cu-Al-Ni alloy belongs to the cubic-to-orthorhombic class [27], which implies that the martensitic phase can exist in six different structural variants and there can be three different twinning systems formed between these variants: Compound, Type 1 and Type 2. In the classical theory of martensitic microstructures[5, 20], the individual variants of martensite are described by different Bain matrices $\mathbf{U}_{1\dots6}$. A given pair of variants (I, J) can then form a twin whenever the middle eigenvalue of the matrix $\mathbf{C} = \mathbf{U}_I^{-1}\mathbf{U}_J^2\mathbf{U}_I^{-1}$ is equal to one.

For the Cu-Al-Ni alloy, each pair of variants can form twins of one or two of the twinning system listed above, and, consequently, there can appear a broad variety of first order laminates (microstructures consisting of parallel, twin-connected plates of two variants of martensite with periodically altering thicknesses [5]) in the single crystals of this alloy. As the Compound twins can cross compatibly with the Type 1 and Type 2 twins, even a broader variety of more complex microstructures can appear by lamination of higher orders.

For compatible connection between a fine microstructure with an effective Bain matrix \mathbf{U}_μ and austenite (described by the Bain matrix equal to the identity matrix \mathbf{I}), it is required that the middle eigenvalue of \mathbf{U}_μ is equal to one. It can be easily shown that this condition is satisfied neither for any of the single variants of 2H martensite, nor for any first order laminate of the Compound twins, while it can be satisfied by the first order laminates of the Type 1 and Type 2 twins with specific volume fractions of the individual components.

As a consequence of this, a single crystal of Cu-Al-Ni austenite, when cooled down, transforms into a fine mixture of first order laminates of Type 1 or Type 2. Then, upon heating, the nucleation of austenite is easily possible due to local compatibility between this microstructure and the nuclei. However, if some mechanical stress is applied onto the martensitic microstructure and the microstructure gets reoriented into either a single variant of martensite, or any other microstructure without $\lambda_2 = 1$, the nucleation of austenite is suppressed, and the reverse transition is shifted to higher temperatures.

The mechanical stabilization appears then at two levels. Firstly, the nucleation barrier is increased by the energy necessary to create the compatible nucleus-to-martensite connection, i.e. to nucleate also the interfacial microstructure. Secondly, the growth of the austenitic nuclei is then accompanied by motion of the interfacial microstructures that dissipates additional

energy and makes the reverse transition more energetically expensive.

The first effect, i.e. the increase of the nucleation barrier, plays an important role for the localization of the first austenitic nuclei. As shown and theoretically explained by Ball et al.[28], in a prismatic sample of the Cu-Al-Ni 2H martensite in a form of a single variant, the nucleation can occur only on one corner of the sample, where the necessary shear strains accompanying the formation of the interfacial microstructure are not blocked by geometrical constraints.

The second effect, i.e. the energy dissipation during the motion of the interfacial microstructure, gives then a possible explanation for the specific morphologies (X - or λ - interfaces, see the next section for more details) observed experimentally in the Cu-Al-Ni single crystals. As shown by Glatz et al.[29], once the nucleus is created these specific morphologies correspond to minimal dissipation paths for the transition from mechanically stabilized martensite to austenite.

3 Experimentally observed interfacial microstructures

3.1 Types of the observed morphologies

The first experimental observation of interfacial microstructures was reported by Basinski and Christian [30, 31] in single crystals of the In-Tl alloy. Two specific morphologies were observed in [30, 31], called X -interfaces and λ -interfaces. The difference between these two morphologies is outlined in Fig.1(a) and (b), where it is shown how the X -interfaces and λ -interfaces provide kinematically compatible connection between austenite and single variant of martensite. Both the X - and λ -morphologies consist of two twinned regions, each including one homogeneous 1st order laminate of twins, and in both cases these two regions meet along one line. In the X -interface, both these regions are connected to the single variant of martensite by a single twinning plane, i.e. the laminate inside each twinned region is parallel to the interface between this region and the single variant of martensite. In the λ -interface, on contrary, the laminate in at least one of the twinned regions is oriented generally with respect to this interface, and so the twinned region is connected to the single variant by a general twinned-to-detwinned planar interface (denoted as *detwinning plane* in Fig.1(b)).

In the Cu-Al-Ni, the first observation of interfacial microstructures was reported by Novák et al.[17]; the observed interface was a λ -interface with clearly visible habit and detwinning planes. However, no analysis of the

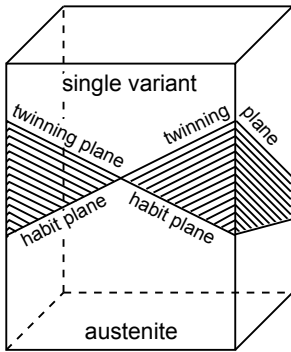
compatibility and full morphology of the interface was provided. Seiner et al.[25, 32] observed first the formation of interfacial microstructures (both X - and λ -interfaces) in thermal gradients and by localized heating, and then also more complex microstructures consisting of so-called *non-classical interfaces* [33]. These microstructures form in the case when the mechanically stabilized martensite entering the transition is not a single variant, but a 1st order laminate of Compound twins that, itself, cannot form any compatible interface with austenite; as a result, the Compound laminate is penetrated by an X - or λ -interface such that a compatible connection appears between austenite and a crossing-twins microstructure (Fig.1(c)). The existence of interfaces providing such a compatible connection was predicted theoretically by Ball and Carstensen [34] prior to the first experimental observations, who also suggested the term *non-classical* for them. The non-classical interfaces exhibit several unique features, for example they can be curved without losing the compatibility if the Compound laminate is heterogeneous.

3.2 Transition front kinetics

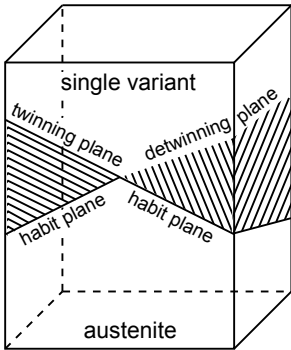
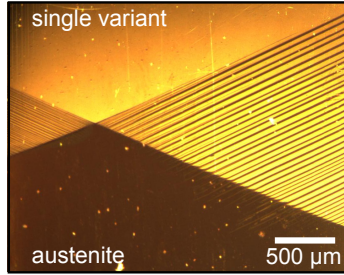
In all reported observations of formation and motion of the interfacial microstructures in Cu-Al-Ni, the austenite \rightarrow martensite transition exhibited a two-stage behavior: after the nucleation in one of the corners of the sample, the growth of the nucleus induced formation of the specific morphology (X - or λ -interface, either classical or non-classical). After this morphology had been formed, the interfacial microstructure propagated through the sample without any changes of its shape. The propagation speed in this stage was always of the order of $1 \text{ mm}\cdot\text{s}^{-1}$ [32], which is probably a velocity determined by equilibrium between the latent heat production and heat conduction (the so-called *Stefan's problem* [35, 36]).

The shape-preserving propagation indicates that the X - or λ - morphologies are in some sense optimal for mobile interfacial microstructures, i.e. that they may exhibit either minimal dissipation (as discussed by Glatz et al.[29]), or minimal pinning on the defects or other local energetic minima (compare with the mobility of macro-twin interfaces discussed in [37]). This significance of the X - and λ -morphologies can be clearly illustrated by monitoring the acoustic emission activity (AE) during the transitions process.

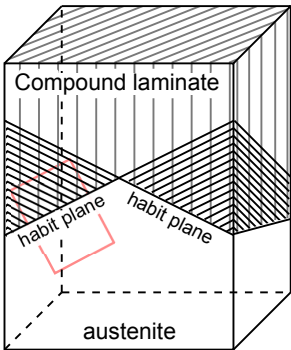
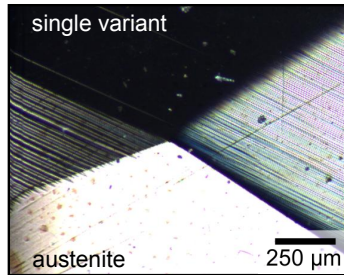
Figure Fig.2(a) shows a simple experimental arrangement for such measurement (see [38] for more details). The sample (a 11 mm long prismatic bar, initially in a single variant of 2H martensite) was freely laid between a heated stage and a cooled stage to ensure the transition front propagation in the prescribed direction and contacted by glycerol droplets to two AE detectors. Such a liquid contacting enabled the detectors to record the AE activity



(a) X - interface



(b) λ - interface



(c) Non-classical interface

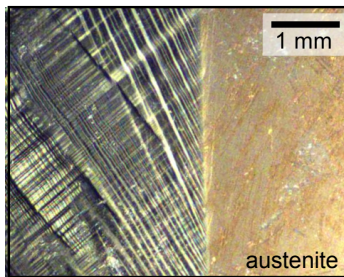


Figure 1: Morphologies of experimentally observed interfacial microstructures: (a,b) X- and λ - interface between austenite and single variant of martensite; (c) non-conventional modification of the X-interface between austenite and a 1st order laminate of Compound twins. The right column shows the sketches of the entire morphologies, the left column show the corresponding optical micrographs. In (c), the location and orientation of the micrograph with respect to the morphology is outlined by the shaded rectangle.

during the whole transition process, regardless of the relatively pronounced changes of the shape of the sample due to the transition. The use of two independent detectors enabled approximate localization of the AE sources, which confirmed that the acoustic signals are indeed generated in the region surrounding the interfacial microstructure.

The results of the AE measurement are seen in Fig.2(b). As soon as the interfacial microstructure is fully formed, the AE rate per second abruptly decreases to nearly zero; another increase appears then in the final stage when the microstructure reaches the free end of the sample and gradually disappears.

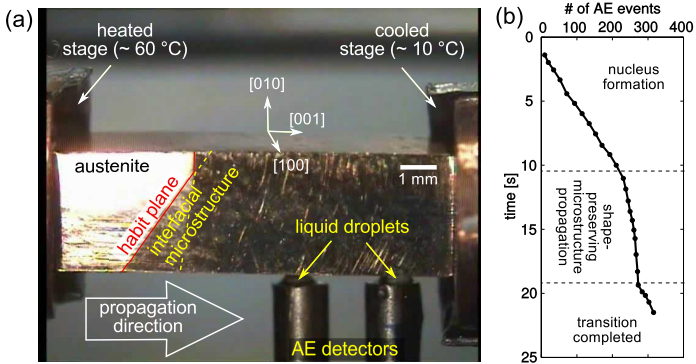


Figure 2: Measurement of the acoustic emission (AE) activity of the interfacial microstructure: (a) experimental set-up with the direction of transition front propagation controlled by a thermal gradient; (b) the AE record. In (a), the mesoscale morphology of the λ -interface is outlined by solid lines (habit planes) and dashed lines (twinning and detwinning planes).

The decrease of the AE rate proves that the propagation of the interfacial microstructure is related to much lower amount of abrupt, burst-like or avalanche-like events than the its formation or its disappearance. As discussed in [25], the mechanisms of motion of the X - and λ -microstructures are based on nucleation and growth of individual martensitic plates at the interfaces between the twinned regions and the mechanically stabilized single variant of martensite. According to the AE measurements this nucleation and growth occurs in a smooth, continuous manner, and is in this sense different from the reorientation processes accompanying the formation of the microstructure and its disappearance. In summary, the AE results are in agreement with the theoretical findings summarized in the next two subsections,

where it is shown that the propagation of an interfacial microstructure cannot be understood as a sequence of energy-minimizing quasi-static states; such sequence would be expected to exhibit significant AE activity when overcoming the energy barriers between the individual minima.

3.3 Compatibility at the meso-scale

Based on the observations of Basinski and Christian [30, 31], the compatibility of X -interfaces in In-Tl was analyzed by Ruddock [39], who showed that such morphology cannot be an energy minimizer, as elastic strains are necessary for it to achieve compatibility. This surprising finding was later confirmed for X - and λ -interfaces in Cu-Al-Ni by Seiner et al.[26] and Glatz et al.[29, 40]; on the other hand, Stupkiewicz and Górzynska-Lengiewicz [41] have shown that from all possible geometric arrangements of the X -interfaces, the experimentally observed ones are those with the lowest elastic energy, i.e. those most close to full kinematic compatibility without elastic strains.

All these calculations are valid at the meso-scale, i.e. at the scale where the laminates inside of the twinned regions can be treated as infinitely fine and are described by homogenized (effective) Bain matrices \mathbf{U}_μ . If the twinned region provides compatible connection between austenite and a single variant of martensite (say the I -th variant described by the Bain matrix \mathbf{U}_I), it is required that the middle eigenvalues of the matrices \mathbf{U}_μ and $\mathbf{C} = \mathbf{U}_I^{-1}\mathbf{U}_\mu^2\mathbf{U}_I^{-1}$ are both equal to one. These conditions must be satisfied for both two twinned regions forming the X - or λ -interfaces or for their non-classical equivalents, for which the Bain matrix of the single variant is replaced by the effective Bain matrix of the Compound laminate $\mathbf{U}_{\text{Compound}}$.

However, these pairwise compatibility conditions onto the planar interfaces are not sufficient for the whole interfacial microstructure to attain full compatibility [5]. In addition, the two twinned regions must be able to form a compatible connection over a line, and, similarly, the single variant (or the Compound laminate) must be able to connect compatibly to austenite over the same line, i.e. the habit planes and twinned-to-detwinned interfaces forming the microstructure must intersect in one line. In Fig.3 (on the right), there is visualized how this condition is satisfied for the X - and λ -morphologies in a simple prismatic bar; in particular, the orientations of the theoretical stress-free compatible interfaces (habit planes, twinning planes and detwinning planes) are shown in this figure. It is clearly seen that these interfaces do not intersect in one line. While for the the λ -morphology the difference is relatively small ($\sim 1.5^\circ$), for the X -morphology a more than 7° misfit is observed [41].

Lastly, besides the conditions for the symmetric Bain matrices, the com-

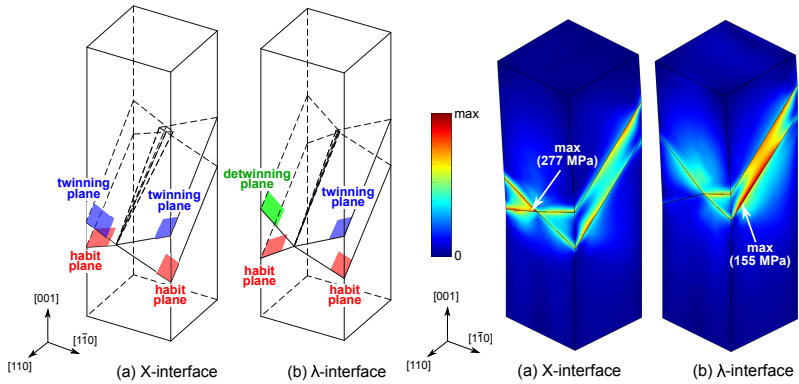


Figure 3: On the left: Misfits between stress-free orientations of the habit planes and twinning or detwinning planes calculated for experimentally observed arrangements of the X - and λ -interfaces. On the right: Tresca stress distribution at surfaces of rectangular samples with X - and λ - interfaces. The arrows indicate the locations of the maxima. (Taken from [40] with courtesy of O. Glatz.)

patibility of the interfacial microstructure gives also strict conditions onto the asymmetric parts of the deformation gradients, i.e. onto the mutual rotations between the individual components of the microstructure. In particular, the mutual rotation between the mechanically stabilized martensite and austenite by compatible connection over the first of the twinned region must be equal to the rotation required for compatible connection over the second twinned region. If this condition is not satisfied, the rotation must be compensated by elastic strains. (The typical rotation misfit for the interfacial microstructures in Cu-Al-Ni is $\sim 0.2^\circ$ [41]).

Fig.3 (on the left) shows the Tresca stress distribution in the X - and λ -morphologies calculated by finite elements method (FEM). The elastic stresses required to compensate the weak incompatibility due to the mismatch of the intersection lines and due to the small uncompensated rotation are obviously concentrated along the habit planes and other interfaces; the maximum values are relatively high (cf. similar calculations for microstructures in Cu-Zn-Al by Balandraud and Zanzotto [42]), higher than plateau-stress for the stress-induced transition in Cu-Al-Ni single crystals or twinning stresses [17], which means that these stresses may locally induce transition or reorientation, and, consequently, modify the morphology at the interfaces. However, such an effect cannot be analyzed at the meso-scale, where the twinned regions

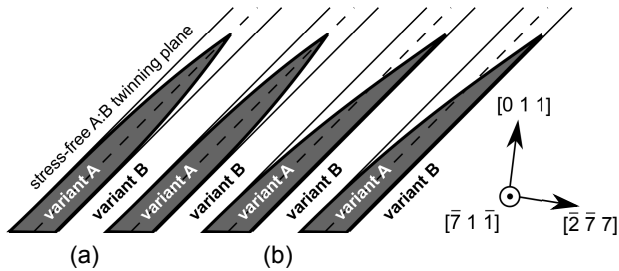


Figure 4: Outline of the difference between the energy-minimizing morphology (a) and the experimentally observed morphology (b) at the detwinning plane of the λ -interface

are treated as homogeneous. A finer experimental and theoretical analysis is required at the level of individual laminae, i.e. at the micro- scale.

3.4 Compatibility at the micro-scale

The meso-scale compatibility conditions require the laminates at the habit planes and detwinning planes to be infinitely fine, which typically leads to branching of the laminates in the vicinity of the interface in order to minimize the energy. This effect has been observed many times for many different alloys and takes partially place also in the interfacial microstructures discussed in this paper. However, a more detailed analysis reveals that there are also other effects, not explainable by the energy minimization.

Most apparently, at the detwinning interfaces between a laminate and a single variant of martensite studied in detail in [43] the laminate exhibits only a negligible amount of branching. Instead, the interface consists of long, bending and tapering needles, and the Bain matrix changes continuously from U_μ to U_I . The elastic strain analysis by FEM presented in [43] revealed, again, that the shape of the needles is not minimizing the energy. The difference between the energy-minimizing shape of the needles and the experimentally observed shape is sketched in Fig.4: while the energy-minimizing morphology is approximately symmetric with respect to the twinning planes of the laminate, in the real (experimentally observed) morphology one of the faces of the needle follows one of the adjacent twinning planes and the thickness of the needle continuously increases until the final volume fraction is reached.

This observed asymmetrization of the morphology may follow either from the shear stresses acting onto the twinned-to-detwinned interfaces due to the weak incompatibility at the meso-scale, or from some requirements on higher

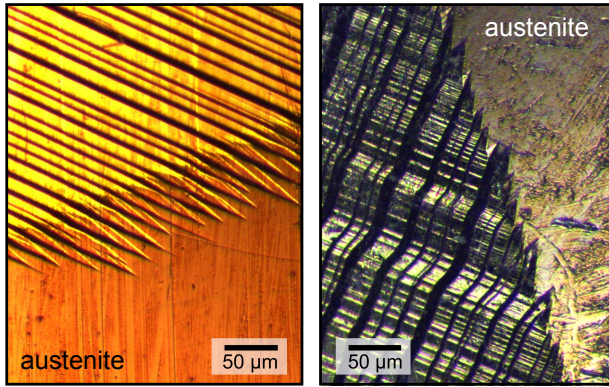


Figure 5: Examples of the needle-like morphologies at habit planes in classical (a) and non-classical (b) interfaces.

mobility of this microstructure - the analysis of the dissipation at such curved interfaces has not been done yet. Nevertheless, similar deviations from theoretically predicted micro-morphologies can be observed also at other interfaces comprised by the interfacial microstructures. Fig.5 shows examples of detailed micrographs of the morphologies at the habit planes ((a) classical habit plane, (b) non-classical habit plane) taken from the experimentally observed X -interfaces. Instead of branching, a needle-like structure is observed again, which has been never reported in the literature for Cu-Al-Ni for forward (austenite \rightarrow martensite) transitions or for reverse transitions without mechanical stabilization. However, such needle-like structure was observed only at some parts of the habit planes and only at sample faces with specific crystallographic orientations, so the understanding to the mechanisms of their formation would require further analysis. On the other hand, this microstructure resembles the zig-zag habit plane morphology observed in Ti-Mn alloy [44] and analyzed theoretically by Stupkiewicz et al [45].

4 Conclusions

The main aim of this paper was to show that the thermally-driven transitions from mechanically stabilized martensite to austenite proceed via formation of specific mobile microstructures, called interfacial microstructures. These microstructures exhibit several weak deviation from the expected energy-minimizing morphologies, both at the meso-scale and at the micro-scale. These deviations, together with the low acoustic emission activity of these

microstructures during their steady-state motion, indicate that the morphology of the interfacial microstructures is probably not governed only by the energy minimization, but also by requirements on mobility.

As mentioned in the Introduction, Cu-Al-Ni is a prototypical SMA, often used to illustrate specific behaviors of this class of materials. In a similar sense, the findings related to the mechanical stabilization effect and to the interfacial microstructures summarized in this paper can be generalized for other SMAs and also for the effective behavior of SMA polycrystals (see e.g. [46] and [47] for such approach adopted for polycrystalline NiTi).

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- 2004 MSc. (Ing.) degree in Physical Engineering, Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague
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Employment:

- 2003 – present Institute of Thermomechanics ASCR, senior researcher (since 2012), deputy-head of the Department of Ultrasonic Methods (since 2011).
- 2008 – present Department of Solid State Engineering, Faculty of Nuclear Sciences and Physical Engineering, CTU in Prague, research and teaching assistant (part time), supervisor of Bc., MSc., and Ph.D. projects.
 - 2005 – 2006 Laboratory of Biomechanics of Man, Faculty of Mechanical Engineering, CTU in Prague, junior researcher (part time).
 - 2005 – 2009 Institute of Physics ASCR, junior researcher (part time).

Scientific stays:

- 2006 Institute of Cybernetics, TTÜ Tallinn, Estonia (2 weeks)
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- 2013 Leibniz Institute IFW Dresden, Germany (6 weeks)

Projects managed (as a principal investigator):

- 2014 – 2015 DAAD Czech-German bilateral project *Mathematical modelling of ferromagnetic shape memory alloys.*
- 2014 – 2016 CSF standard project *Experimentally justified multiscale modelling of shape memory alloys.*
- 2009 – 2010 CSF postdoc project *Elasticity of Martensitic Microstructures.*
 - 2007 pilot project of IT ASCR *Dynamical formation and mobility of microstructures in single crystals of shape memory alloys.*
 - 2006 internal project of CTU *Development of the inverse procedures for resonant ultrasound spectroscopy (RUS) for generally oriented, non-rectangular samples.*

Publication record (SCOPUS database): 55 documents, 186 citations without self-citations, h -index = 11.

Selected journal papers (the IF-value relates to the publication year):

1. Seiner, H., Ramirez, C., Koller, M., Sedlák, P., Landa, M., Miranzo, P., Belmonte, M., Osendi, M.I. Elastic properties of silicon nitride ceramics reinforced with graphene nanofillers (2015) *Materials and Design*, 87, pp. 675-680. **(IF=3.501)**
2. Kruisová, A., Seiner, H., Sedlák, P., Landa, M., Román-Manso, B., Miranzo, P., Belmonte, M. Acoustic metamaterial behavior of three-dimensional periodic architectures assembled by robocasting (2014) *Applied Physics Letters* 105, art. no. 211904. **(IF=3.515)**
3. Kabla, M., Seiner, H., Musilova, M., Landa, M., Shilo, D. The relationships between sputter deposition conditions, grain size, and phase transformation temperatures in NiTi thin films (2014) *Acta Materialia*, 70, pp. 79-91. **(IF=3.940)**
4. Seiner, H., Straka, L., Heczko, O. A microstructural model of motion of macro-twin interfaces in Ni-Mn-Ga 10 M martensite (2014) *Journal of the Mechanics and Physics of Solids*, 64 (1), pp. 198-211. **(IF=4.289)**
5. Seiner, H., Kopeček, J., Sedlák, P., Bodnárová, L., Landa, M., Sedmák, P., Heczko, O. Microstructure, martensitic transformation and anomalies in c' -softening in Co-Ni-Al ferromagnetic shape memory alloys (2013) *Acta Materialia*, 61 (15), pp. 5869-5876. **(IF=3.940)**
6. Seiner, H., Sedlák, P., Koller, M., Landa, M., Ramírez, C., Osendi, M.I., Belmonte, M. Anisotropic elastic moduli and internal friction of graphene nanoplatelets/silicon nitride composites (2013) *Composites Science and Technology*, 75, pp. 93-97. **(IF=3.633)**
7. Heczko, O., Straka, L., Seiner, H. Different microstructures of mobile twin boundaries in 10 M modulated Ni-Mn-Ga martensite (2013) *Acta Materialia*, 61 (2), pp. 622-631. **(IF=3.940)**
8. Seiner, H., Bodnárová, L., Sedlák, P., Janeček, M., Srba, O., Král, R., Landa, M. Application of ultrasonic methods to determine elastic anisotropy of polycrystalline copper processed by equal-channel angular pressing (2010) *Acta Materialia*, 58 (1), pp. 235-247. **(IF=3.781)**