Full length article

Modulated formation of lath martensite: Influence of uniaxial compressive load and transformation-induced plasticity

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ABSTRACT

The austenite → martensite transformation occurring upon cooling of an FeNiCoMo alloy, similar to commercial FeNiCoMo maraging steels, thereby forming lath martensite, was investigated by dilatometry with regard to the influence of an externally applied compressive load on the peculiar transformation behavior in this system, which exhibits a step-wise mode of transformation upon slow cooling, as shown recently. Temporary, external loading during isothermal interruption of the continuous cooling at different stages of the transformation revealed a decreasing degree of plastic deformation proportional with the decreasing amount of retained austenite, implying that the plastic deformation is predominantly accomplished within the austenite phase. Upon resuming the cooling, the step-wise transformation was found to be unaffected. Application of a constant, comparatively high external load during continuous cooling caused a step-wise length *decrease* in the temperature range of the transformation instead of the step-wise length *increase* observed for the martensitic transformation in absence of an external load. The plastic deformation happening during the transformation occurs also for loads below the yield limit of pure austenite. This genuine transformation-induced plasticity was ascribed to the generation of mobile dislocations within the austenite upon the formation of martensite, thereby enabling plastic deformation in addition to the intrinsic deformation induced by the load for a specimen in the absence of transformation. Because the transformation into martensite also during loading takes place in a step-wise manner, the generation of these mobile dislocations occurs step-wise too, thus leading to the observed step-wise plastic deformation and corresponding step-wise length decrease during the transformation.

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1. Introduction; energetic considerations; martensite formation with and without external loading

Recently, a regularly modulated austenite → martensite transformation behavior has been observed for different lath martensites (i.e. several FeNi alloys and an FeNiCoMo alloy [1–3]): a reproducible series of transformation-rate maxima occurring upon cooling, detected both by use of high-resolution dilatometry and of calorimetry [1,2]. An understanding of this phenomenon was obtained recognizing the evolution of the hierarchical microstructure of lath martensite:

Upon transformation to martensite, the initial austenite grains are subdivided in packages of parallel blocks. As lath martensite (α', bcc) and austenite (γ, fcc) usually show an orientation relationship close to the Kurdjumov-Sachs (K–S) orientation

relationship (OR) [4,5], 24 austenite-martensite orientation variants for four possible pairs of $\{111\}_\gamma||\{110\}_{\alpha'}$ planes, each with six combinations of the directions $<1{-}10>_\gamma||<{-}111>_{\alpha'}$, exist [6]. All blocks within one package have the same pair of $\{111\}_\gamma||\{110\}_{\alpha'}$-planes and each block consists of sub-blocks of two specific orientation variants, each sub-block built by martensite laths of one orientation variant with only small mutual misorientation [7,8].

The emergence of a modulated transformation behavior upon formation of this special microstructure is explained as follows: Generally, martensite formation upon cooling is controlled by the interplay of the release of chemical Gibbs energy, serving as driving force for the transformation, and the developing strain energy, which (at first sight) opposes progress of the transformation

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[9–11].[1] Considering the contributions of these energies on the block level, i.e. for the formation of a single block in one package, a block grows in width whereupon the increasing deformation energy, introduced into the adjacent material due the shape strain associated with the formation of martensite in austenite, reduces the net driving force. As a consequence, growth of the block slows down, and, depending on the rate of provision of chemical driving force, i.e. depending on the cooling rate, can virtually come to a halt. Part of the deformation energy stored in the adjacent material can then be released by the formation of a new block of another OR variant set (out of the 3 possible sets of 2 sub-block OR variants [7,8,13]), because of the partial accommodation of the shape strain associated with formation of this block [13–15]. This (to be) released part of the deformation energy then serves as an additional driving force for the formation of the next block. Thus, upon further cooling, i.e. provision of additional chemical driving force, the formation of a new adjacent block of a different OR variant set is favored over further growth of the current block. Hence, at one point upon further cooling, the nucleation barrier for formation of this next block can be overcome and a new block of the package is formed. The resulting transformation rate as a function of the temperature upon cooling has been modeled in Ref. [3] on a package level. The *macroscopically* observable transformation-rate maxima are then explained by a (more or less) simultaneous formation of all next blocks in all packages, where realization of such simultaneity is thought to be a consequence of (more or less) identical local conditions for subsequent block formation in all packages, realized by a thermally activated process, for instance by local stress relaxation [1–3]. This requires a cooling rate slow with respect to the rate of the thermally activated relaxation.

In the above model description, the development of *local* elastic and/or plastic strains (in the material adjacent to the growing block) plays a cardinal role for the occurrence of the modulated transformation behavior of lath martensites. Hence, it is interesting to investigate the effect of an *externally* applied load on the (emergence of the) transformation-rate maxima.

Depending on the magnitude of the applied force, different effects of external loading on the martensitic transformation mechanism can be observed (e.g. Refs. [16–18]). If the applied load is smaller than the yield strength of the material, only elastic deformation of the material occurs: An externally applied uniaxial force then acts as an additional mechanical driving force for certain favorably oriented martensite OR variants and assists the formation of martensite, thus leading to an increase of the martensite start temperature M_s. This is denoted as stress-assisted martensite formation [17,18].

An externally applied stress exceeding the yield strength of the material causes plastic deformation, i.e. introduces additional dislocations, which are often thought to provide new, more potent nucleation sites[2] [20], leading to an increase of the M_s temperature. This is termed *strain-induced* martensite formation [17,21]. It is noted that if the applied stress is large enough to exceed the yield limit, both *stress-assisted* and *strain-induced* martensite formation can operate, and both effects will lead to an increase of M_s.[3]

It should be recognized that large externally applied stresses causing plastic deformation can also hinder the martensitic transformation, if the amount of dislocations introduced is that large that the martensite/austenite interface becomes mechanically stabilized (e.g. by forest dislocations [24]): the movement of the martensite/austenite interface is then impeded by the resistance of these dislocations against glide [25–27], and consequently the M_s decreases and also continued transformation is hindered.

The above discussion illustrates that upon loading, various processes can significantly influence the generation and/or activation of martensite-nucleation sites. Against this background, in the present work the effect of external loading on the recently discovered modulated lath-martensite formation behavior [1] was investigated in order to expose the operating transformation mechanisms.

2. Experimental

2.1. Specimen preparation

The present project on the effect of loading on martensite formation was carried out using an Fe-18.5 wt% Ni −9 wt%-Co 5 wt% Mo alloy which showed the unusual transformation-rate modulation very distinctively upon applying cooling rates up to 2.5 K min^{-1} (see Ref. [1]). This alloy is comparable with typical FeNiCoMo maraging steels, but without additional elements forming intermetallic phases. An FeNiCoMo alloy rod was prepared by induction melting from elemental Fe (4N), Ni (4N6), Co (3N) and Mo (3N) with a diameter of 12 mm and a length of 15 mm. After homogenization for 24 h at 1323 K (i.e. within the austenitic regime, with $A_f \approx 1150$ K) under Ar atmosphere and quenching in ice water, the rod was hammered down to a diameter of 6 mm and cut into pieces of approximately 10 cm. These pieces were again annealed for 24 h at 1323 K and subsequently quenched in ice water. Finally, cylindrical dilatometer specimens were machined with a diameter of 5 mm and a length of 10 mm. The exact chemical composition of the alloy was determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and the degree of carbon contamination was determined by a combustion method (see Table 1).

2.2. Dilatometric measurements under constant uniaxial load

To measure the length change as a function of time/temperature and under applied uniaxial, compressive force, a differential deformation dilatometer with inductive heating of the specimen (Bähr DIL 805D), employing a quartz measurement system, was used The specimen is mounted between two quartz deformation stamps (see also [28]); the relative distance of the stamps can be determined with an accuracy of 50 nm [29,30]. For monitoring the temperature, two thermocouples were spot-welded onto the specimen surface, one at half length of the cylindrical specimen, used for controlling the temperature, the other one close to one of the flat faces of the cylindrical specimen. The inductive heating principle inadvertently leads to a temperature gradient close to the

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[1] The contribution of the interface energy can generally be neglected in comparison to the strain energy contribution (e.g. see Ref. [12]).

[2] Nucleation of martensite is often described by a faulting process derived from defects existing in the initial austenite phase [10,11,19]. However, it has been argued in Ref. [20] that dislocations don't act themselves as nucleation sites but that their presence is necessary to initiate slip for shape accommodation around nucleation sites which are provided by the grain boundaries. Nevertheless, *additional* dislocations facilitate the transformation in this alternative concept, as well.

[3] Stress-assisted martensite formation in steels is often considered to be dominating even if the external load exceeds the yield strength of the austenite [22,23].

Table 1
Chemical composition of the FeNiCoMo alloy specimens determined by ICP-OES (Ni, Co, Mo) and combustion analysis (C).

	Fe	Ni	Co	Mo	C
wt%	balance	18.7 ± 0.2	9.10 ± 0.09	5.11 ± 0.05	$(2.9 ± 0.2)*10^{-3}$
at.%	balance	18.4	8.94	3.08	$14.0*10^{-3}$