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Grain orientation dependence of phase transformation in the shape memory alloy Nickel–Titanium

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Abstract—The dependence of grain orientation on stress-induced martensitic transformation in superelastic, polycrystalline Nickel–Titanium sheet was examined at the microstructural length scale. Full-field strains, indicative of transformation extent, were characterized in fields of view of nominally 100 μm × 100 μm using a custom combination of scanning electron microscopy with distortion-corrected digital image correlation. It was found that similarly oriented grains do not necessarily transform similarly, in contrast to a common assumption in mean-field theories. Specifically, grains with similar orientation (as determined by the misorientation of the grain and specimen axes) showed variation in both the mean strain of the grain as well as the range (heterogeneity) of strain across the grain, as determined from surface measurements. Additionally, neither grain size nor degree of misorientation (of common crystal axes from the loading axis) affected the mean strain and strain range.

Keywords: Nickel Titanium; Superelasticity; Scanning electron microscopy; Digital image correlation; Mean field theory

1. Introduction

Nickel–Titanium (NiTi) alloys belong to a class of materials commonly referred to as shape memory alloys (SMAs). The defining characteristic of SMAs is the ability to recover strains as large as 10% through a solid-to-solid, diffusionless phase transformation [1]. In the case of NiTi, this transformation takes place between a high symmetry cubic (B2) austenite and one or more monoclinic (B19′) martensite variants [1–4]. While the crystallography and thermomechanical properties of this transformation are well understood for single crystals [2,5–7], the mechanisms of transformation are unclear in polycrystalline SMAs. Mean-field and phenomenological models of phase transformation [8–15] use properties derived from observing single crystal behavior and assumptions about the similarity and completeness of martensite transformation in grains of similar orientation to predict macroscopic stress–strain curves of polycrystalline SMAs. While these predictions are reasonable approximations of averaged macroscopic behavior under simple loading conditions, their underlying assumptions have not been validated and may not be widely applicable. Recent experimental work using neutron-diffraction techniques [16] has shown that the influence single crystal parameters like transformation Schmid factors are overshadowed by the influence of grain neighborhood. Furthermore, recent theoretical work suggests that martensitic transformation in polycrystals can take on complex and heterogeneous configurations when the constraints of neighboring grains and interactions with other deformation mechanisms (such as plasticity) are taken into account [17]. The aim of this work is to examine the assumptions used in many mean-field theories that grain orientation is sufficient to predict the magnitude of martensite transformation strain and that grains transform fully.

Transformation strain and habit plane formation in NiTi single crystals have been identified experimentally [2,7,18–20] and modeled using phenomenological theory of martensite transformation [13,21,22] for both tensile and compressive loading. Under tensile loading [2,7,18], the experimentally observed transformation strain was consistent with a single correspondent variant pair (CVP) completely replacing the parent austenite volume at the end of the transformation plateau. Additionally, martensitic variants that dominated the transformation were identified by the delineation of specific habit planes on the sample surface [2,4,18] and matched well with predictions based on the Schmid factor for each variant. For a single crystal of Nickel–Titanium under uniaxial tension, the total strain accommodated by the martensite for an arbitrary crystal orientation could be accurately predicted [2,23]. Characterization of single crystals under compression [6,20] has also been performed. However, because non-local transformation of bulk NiTi occurs under

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compression, no defined habit plane is formed, making macroscopic identification of the particular active CVPs difficult. Rather, these studies focused on the critical resolved shear stress required for martensitic transformation and on the relative activity of plastic deformation and transformation. For many grain orientations, a similar level of plastic deformation and martensitic transformation under compression made attribution of measured strain to one of these mechanisms more difficult than in the simple tension case, where the stress required for transformation is well below that required for plastic deformation [20]. However, in grains with orientations where plasticity was significantly suppressed, compression results provided confirmation of transformation strains predicted by the phenomenological model for single crystals. More recently, single crystal experiments performed on micropillars in compression have allowed for the confirmation of exact martensite variants active under compressive loads [24–28]. At the microscale bulk plasticity is suppressed, and in solutionized samples individual CVPs can form with distinct and measureable habit planes [24]. Under these conditions the formation of specific martensite variants can be determined by habit plane measurement and the results can be compared directly against phenomenological predictions [23]. When combined with bulk single crystal results, the confirmation of specific active CVPs in micro compression validates the predictive power of phenomenological models for uniaxial loading of single crystals of arbitrary orientation.

The results of single crystal experiments are used to extend predictions of transformation strain and martensite variant selection to polycrystalline NiTi [14,22,29–32]. In polycrystalline NiTi, the constraints imposed by neighboring grains complicate the stress states of individual grains, which can no longer be described by the uniform global stress state. One approximation used to circumvent the complications of such constraints is to consider a polycrystalline solid as an averaged collection of single crystals, with orientations determined by the orientation distribution function (ODF) [22,30,31,33]. An estimate of the total recoverable strain for shape memory [31] or superelasticity [22,30,33] can then be made by averaging the contribution of each independent austenite grain by assuming complete transformation to martensite. By choosing the most favorable single variant of martensite, this averaging produces an upper bound that can over estimate the bulk strain in polycrystalline samples by as much as 100% of the experimentally measured strain [30,33]. However, this approach does capture trends, such as the decrease of maximum accommodated strain in textured, rolled material when tensile deformation occurs perpendicular rather than parallel to the rolling direction. An improved estimate can be made by considering the most favorable CVP—rather than the individual variant—of martensite [23,30]. While this calculation better matches the recovered strain magnitude, the variation in transformation strain shows less sensitivity to sample orientation and is still an overestimate that ignores the constraints imposed by neighboring grains when compared with experimental data. A further refinement can be made by considering the Taylor bound of accommodated strains for single crystals, in which multiple CVPs of martensite are allowable [30,32,34]. This last consideration includes compatibility requirements at representative grain boundaries and most closely matches experiment, but again much of the orientation dependence is lost, resulting in an underestimate of the recoverable strains in polycrystalline specimens.

Although the incorporation of incomplete transformation and alternate deformation modes such as bulk plasticity and dislocation motion have been proposed to improve model accuracy, the underlying assumptions of applying single crystal response to polycrystals require further investigation. Combined single crystal responses do not accurately predict the transformation strain of polycrystalline specimens. Phenomenological models assume a single transformation path—of either a single variant, variant pair, or variant subset—to consume the entirety of a crystal oriented along a particular axis. Measuring the details of this phase change directly is difficult because of the small length scale of the twinned martensite microstructure. However, each transformation path imparts a known strain based on the phase fraction of each martensite variant in the final configuration. While it is possible for different transformations to accommodate the same strain, a particular martensite volume fraction/configuration pair cannot simultaneously maintain different strain levels. Thus, while strain measurement alone cannot confirm that similarly oriented grains have transformed to the same configuration of martensite or to the same volume fraction of the same variants, it can pin point at what strain and the specific locations (grains) where *different* martensite configurations form.

In the present study, an investigation of a principal assumption in most mean-field and phenomenological theories—that similarly oriented grains in polycrystalline SMAs transform similarly—was conducted using structural information from electron backscattered diffraction (EBSD) linked to local strain data provided by digital image correlation of scanning electron micrographs (SEM-DIC). This approach enabled grain-to-grain comparisons of transformation strain and grain orientation which satisfied three principal criteria: First, that strain, phase, and orientation data can be quantitatively compared, either within the same measurement or between multiple measurements, to associate microstructural features with martensite transformation and recovery. Second, that the approach has sufficient resolution relative to the microstructural features (e.g. parent austenite grains) to allow multiple aspects of the martensite transformation (i.e. overall transformation strain and transformation heterogeneity) to be compared across the sampled microstructure. Lastly, that a single measurement captures strain (or phase) data equally well for martensite and austenite phases. This last requirement is difficult to address experimentally, given the disparate size scales of parent austenite and martensite microstructures. Previous studies using diffraction-based techniques [35,36] were the first to achieve the necessary resolution to examine martensitic transformation on the microstructural length scale. However, the fineness of martensite twins required that martensite transformation be inferred from the absence of austenite diffraction patterns. Additionally, local strain measurements were limited to the elastic strain of the austenite phase without a similar local measurement of transformation strain in martensite. Optical microscopy [37] has been successfully used to resolve the fine martensite surface structure, but did not link this data to local strains. The experimental approach used here tracked the local strains of both phases with high spatial resolution and accuracy across large fields of view, in order to determine if similarly oriented grains do or do not transform