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Development of high throughput assays for establishing process-structure-property linkages in multiphase polycrystalline metals: Application to dual-phase steels

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A B S T R A C T

Process-structure-property (PSP) linkages are central to the development and deployment of advanced materials in emerging technologies. Conventional approaches for establishing these are generally highly customized, qualitative, and demand major investments of time and effort. In this paper, we formulate novel, data-driven, high throughput, assays for exploring PSP linkages in structural metals. These assays are built on recent advances in low-dimensional quantification of material structure using spatial correlations and principal component analyses, as well as in the mechanical characterization using spherical nanoindentation. These novel protocols are demonstrated on a dual phase (DP) steel that exhibits rich multiphase polycrystalline microstructures.

1. Introduction

A great deal of attention in materials innovation is now focused on the accelerated deployment of advanced materials in commercial products [1–6]. An important strategy for realizing this ambitious goal is the development, validation, and adoption of high throughput assays for rapid exploration of the extremely large materials and process design spaces involved. Indeed, significant effort in recent years [7–21] has been aimed at extracting high value (most useful for a selected application) process-structure-property (PSP) linkages [22–34] using a variety of approaches (including multiscale experiments, multiscale models, and data analytics).

Our focus in this paper will be on the protocols needed for the accelerated, experimental, exploration of PSP linkages in multiphase structural metals and alloys (e.g., dual-phase or DP steels). The processing histories employed on these materials typically involve a sequence of thermo-mechanical treatments, whereas the main properties (or performance characteristics) of interest are largely related to the plastic response of the material. Since one typically employs standard tensile tests [35] for evaluating the plastic properties of the material, it becomes necessary to make relatively large quantities of material samples with statistically homogeneous microstructures throughout the gage section (of the sample used in the tension test) for each combination of material chemistry and process path studied. This requirement drives up the cost and effort substantially, as is evident from some of the recent innovative efforts in this direction [12]. In other words, if it were possible to measure reliably the plastic response of the material in very small volumes, it would become possible to reduce dramatically the cost and effort involved in rapid screening of large design spaces in material chemistry and process histories.

Indentation has been employed extensively in prior literature [36–52] for evaluating the mechanical response of materials in small volumes. However, the use of this technique has been restricted largely to estimation of modulus and hardness. Only recently, it has been shown that it is possible to extract meaningful indentation stress-strain (ISS) curves directly from spherical indentation through a rigorous set of data analyses protocols. These new protocols have thus far been demonstrated mainly at the very small length scales (indentation zones much smaller than a single grain) [53–60]. There is, however, substantial promise for their application at the meso-length scales (where the indentation zone covers several grains [61]).

Beyond the measurement of mechanical response in small

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volumes, the next major impediment in extracting PSP linkages in advanced structural metals comes from the lack of a rigorous, yet practical, framework for the statistical quantification of their rich microstructures. Most of the currently employed approaches [38,45,47,48,51,62–67] are based on highly simplified measures such as elemental compositions, phase volume fractions, average grain sizes of constituent phases, and the orientation distribution function (ODF). Clearly, the number of distinct microstructures one can reconstruct while meeting specific targets on these measures is extremely large, suggesting that this set of measures is likely to be inadequate for capturing all of the salient features of the microstructure. More formally, one might recognize most of the measures listed above (except the average grain size) as 1-point statistics of the microstructure in that they do not capture the morphological attributes of the microstructure. Indeed, one can employ the more advanced 2-point statistics (formally called the 2-point spatial correlations) [30,31,68–75] to arrive at a more rigorous quantification of the material microstructure. However, most of the prior examples of the application of the framework of 2-point statistics have been limited to multiphase materials, with only a few focused on the application to polycrystalline materials [76–78]. The main difficulty of applying the 2-point statistics measures to polycrystalline samples arises from the very large number of local states present (for example, each distinct grain orientation encountered in the sample needs to be treated as a distinct local state), and the fact that the corresponding number of the distinct 2-point statistics is exceedingly large. In addition to being multiphase and polycrystalline, the microstructures of advanced alloys such as DP steels exhibit grain-scale heterogeneity in the spatial distribution of the dislocation densities (see Fig. 1). Not surprisingly, a rigorous quantification of these complex microstructures is not readily accomplished with the currently available toolsets. Clearly, one needs to make suitable simplifications in order to establish practically useful PSP linkages for advanced structural metal alloys.

The main goal of this work is to explore the viability of high throughput experimental assays for establishing PSP linkages in multiphase polycrystalline metals, while utilizing small sample volumes and leveraging some of the recent advances described earlier (i.e., spherical microindentation stress-strain protocols and the framework of 2-point statistics). We have selected DP steels for this study because of their importance to several advanced technologies, owing to their excellent combination of continuous yielding behavior, high tensile strength, high work-hardening rate, and good ductility [38,41,45,47,51,62,63,66,79–87]. These properties are generally achieved in DP steels through a special heat treatment process called intercritical annealing [36,52,64,65,67,80,84,88] during which the material is heated up to the austenite/ferrite region, held for a certain amount of time, and quenched to room temperature. Intercritical annealing results in formation of hard particles of martensite (α') in a soft matrix of ferrite (α) grains. This step is usually followed by additional cold work and heat treatment called bake hardening (BH [50,66,79,82,85–96]; cf. Fig. 1) to achieve the desired combination of mechanical properties. The main mechanisms involved in this overall thermomechanical process are as follows: (i) Introduction of mobile dislocations in ferrite grains at the vicinity of α/α' interfaces ([47,62,67,79,82,83,90,97–99]; see Fig. 1(b)), generally attributed to the volumetric plastic strains induced during the austenite to martensite transformation that occurs during the quenching from

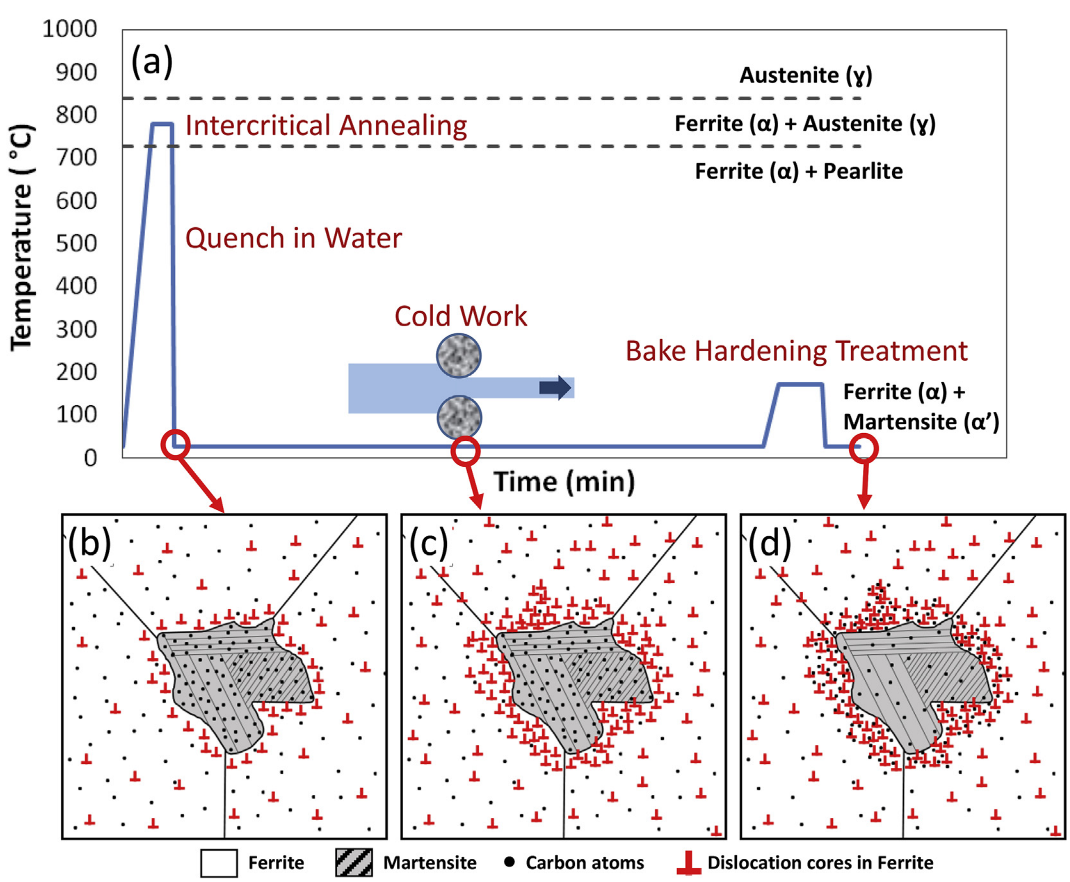

Fig. 1. Schematic of a dual phase steel processing path and the expected strengthening mechanisms in each step of the thermo-mechanical process.