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Predicting flow curves of two-phase steels from spherical nanoindentation data of constituent phases: Isostrain method vs. non-isostrain method

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ABSTRACT

A procedure is suggested to predict the flow curves of two-phase steels using data from nanoindentation experiments performed with two spherical indenters having different radii. The procedure incorporates two steps: First, the "macroscopic" (or size effect corrected) stress–strain relations of each constituent phase are estimated based on the concepts of indentation stress/strain and indentation size effect. Then, the "overall" (or composite) flow curve of two-phase steel is extracted in two different ways; an isostrain method (ISM) and a non-isostrain method (NISM). The appropriateness of the proposed procedure was examined by performing a series of spherical nanoindentation tests on various two-phase steels (consisting of ferrite–pearlite or ferrite–bainite). Reasonable accuracy of the prediction was validated by comparing the predicted curves to the tensile curves obtained from standard tests of bulky samples. In addition, interestingly, the predictions made by the simple ISM were almost identical to those by the more sophisticated NISM, though the NISM used more realistic assumptions.

1. Introduction

In the development of advanced structural alloys, controlling their microstructure is undoubtedly the most important aspect to optimize mechanical performance. Extensive research has been performed to establish proper models for predicting microstructures from chemical compositions and processing conditions, and now some models are practically applied in the industrial fields (especially, steel industries). However, despite significant effort, there is no established way to precisely predict the final mechanical properties from microstructural features.

Most previous research on the prediction of mechanical properties (Asgari et al., 2009; Choi et al., 2009a; Hüper et al., 1999; Ishikawa et al., 2000, 2006a,b; Karlsson and Linden, 1975; Khan et al., 2012; Kim and Thomas, 1981; Koo et al., 1980; Lanzillotto and Pickering, 1982; Ramazani et al., 2012, 2013a,b; Rudiono and Tomota, 1997; Sun and Wagoner, 2013; Tomota et al., 1976, 1992; Zhu and Lu, 2012) has targeted the development of a proper model for the alloys consisting of the two-phase microstructure that is common in many commercial-grade steels. It is well accepted that the nature of plastic flow behavior in two-phase steels depends on the mechanical properties of each phase and the volume fractions of the phases in many different ways (Choi et al., 2009a; Kim and Thomas, 1981; Koo et al., 1980; Lanzillotto and Pickering, 1982). Therefore, in developing a prediction method for designing two-phase steels and optimizing their flow

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properties, both the properties and fractions of the phases and the way how they affect the overall flow curve should be carefully taken into consideration.

While one of the simplest approaches for predicting the flow curve of two-phase steel is a method based on the assumption of equal strain in each phase (hereinafter called "isostrain method", ISM), there is a more sophisticated method that assumes different strain in each phase (hereinafter referred to as "non-isostrain method", NISM). One of the most popular NISMs was suggested in the middle of 1970s by Tomota and colleagues (Rudiono and Tomota, 1997; Tomota et al., 1976, 1992) who designed a micromechanical way on the basis of Eshelby's inclusion theory (Eshelby, 1957) and Mori–Tanaka mean field concept (Mori and Tanaka, 1973). Ishikawa and colleagues (Hüper et al., 1999; Ishikawa et al., 2000, 2006a,b) adopted Tomota's NISM to optimize the volume fractions of the phases in high-strength dual-phase steels. However, a fundamental difficulty remains in previous ISM and NISM; although the flow curves of each phase are essentially required for both methods, standard tensile/compression tests cannot be performed on such a small volume of micro-phase. One promising technique to overcome this difficulty is nanoindentation (Oliver and Pharr, 1992, 2004) which has been widely used to probe local mechanical properties in crystalline/amorphous metals (for instance, Choi et al., 2009b; Chollacoop and Ramamurty, 2005, 2006; Jang et al., 2007; Moon et al., 2008; Oh et al., 2011; Yoo et al., 2009) because the technique requires only a small volume of target material. While three-sided pyramidal indenters (especially, Berkovich indenter having a centerline-to-face angle of 65.3°) have been popularly adopted for the technique, nanoindentation with a spherical indenter provides distinct merits (Choi et al., 2009a, 2011; Jang et al., 2005; Johnson, 1985; Kang et al., 2013; Tabor, 1951; Yoo et al., 2010) based on the fact that stresses and strains beneath the contact increase as penetration depth (and load) increases. With continuum mechanics viewpoints, similar behavior does not occur during a sharp indentation due to the tip's geometrical self-similarity.

In the present study, we suggest a procedure for predicting the overall flow curves of two-phase steel from nanoindentation data obtained with two spherical indenters having different radii. Two different methods, including ISM and NISM, were used for the predictions, and the difference in their results was investigated. The appropriateness of the proposed procedure was examined by comparing the predictions to the experimental data obtained from standard tensile tests of bulky samples. Before starting, it is noteworthy that, here, the term "two-phase" indicates two different microstructures such as ferrite vs. pearlite or ferrite vs. bainite. Although pearlite and bainite are not truly "phases" but "specific microstructures", (Rudiono and Tomota, 1997) they are treated as phases in the present study for the sake of simplicity.

2. Experimental

The two-phase steels examined in this study were three low-carbon steels and an API X100 linepipe steel. The low-carbon steels having compositions (in wt%) of Fe–*X*%C–1.2Mn–0.15Si (with *X* = 0.04, 0.07, and 0.10) were heated up to 1000 °C for 600 s and then cooled down to room temperature at 1 °C/s using a thermo-mechanical simulator Gleeble 1500 (Dynamic Systems Inc., Poestenkill, NY) in order to obtain two-phase microstructures of ferrite and pearlite. The API X100 steel consisting of ferrite–bainite microstructures was a commercial-grade high-strength steel having a nominal composition of Fe–(0.05–0.07)C–0.25Si–2.0Mn–0.01P–0.001S–0.05Nb–0.05V–0.3Mo (in wt%).

All specimens were mechanically polished with fine SiC paper with grit number of up to 2000, then electrolytically polished using a Lectropol-5 instrument (Struers, Westlake, OH) in a solution (ethanol 80%, distilled water 14%, perchloric acid 6%) to avoid artifacts related to a hardened surface layer possibly introduced during grinding. Specimens were etched with 3% Nital solution for microstructure observations by an optical microscope (Olympus, Tokyo, Japan). The grain size and volume fraction of each phase were measured by an image analyzer, Image-Pro (Media Cybernetics Inc., Silver Springs, MD).

Nanoindentation tests were carried out using a Nanoindenter-XP (formerly MTS; now Agilent Technologies, Oak Ridge, TN) with two spherical diamond indenters having different radii. The real (or "effective") tip radius R was determined by analyzing data from the indentations made on a fused quartz sample based on Hertzian contact theory (Johnson, 1985). Load-controlled indentations were performed at a peak load P_{max} of 15 mN under a constant indentation strain rate $(dP/dt)/P$ (where P is the indentation load) of 0.05/s. After indentation, the specimens were slightly etched in 3% Nital acid for examining whether or not the indentation was made inside the target phase in a field-emission SEM, JSM-6330F (JEOL Ltd., Tokyo, Japan). Finally, for comparison purposes, the uniaxial stress–strain curves were obtained through standard tensile tests that were conducted using a universal testing machine, Z100 (Zwick GmbH & Co., Ulm, Germany).

3. Procedure for predicting the flow curve of two-phase steel

3.1. Estimating macroscopic flow curves of constituent phases

The procedure suggested here for predicting the flow curve of two-phase steel incorporates two steps; (1) experimental estimation of the "macroscopic" flow curves of each constituent phase through spherical nanoindentations, and (2) theoretical extraction of the "overall" (or composite) flow curve of two-phase steel from the curves of each phase.

The main part of the first step is to convert the spherical nanoindentation data to the macroscopic stress–strain curve of each phase. Note that the macroscopic curve means the size effect corrected curve that can be directly compared to the curve obtained from the standard tensile test on a bulky sample. It is well accepted (e.g., see Cao and Lu, 2004; Herbert et al., 2006;