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Specimen size effects on the weakening of a bulk metastable austenitic alloy

Chansun Shin [a,*], Sangyeob Lim [b], Hyung-ha Jin [b], Peter Hosemann [c,**], Junhyun Kwon [b]

[a] Department of Materials Science and Engineering, Myongji University, Yongin 449-728, Republic of Korea
[b] Nuclear Materials Research Division, Korea Atomic Energy Research Institute, Daejeon 305-353, Republic of Korea
[c] Department of Nuclear Engineering, University of California, Berkeley, CA 94720, USA

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ABSTRACT

In this work, we are investigating the scaling effects on an austenitic stainless steel with changing grain size using microcompression testing. It is our aim to evaluate at what sample-to-grain size ratio the mechanical properties such as yield stress deviate from the macroscopically determined data. It was found that decreased yield stresses with decreasing specimen size (weakening) occur during the microcompression. The weakening was observed when the diameter-to-grain size ratio (D/d) dropped below a critical value. The effect of the grain size on the critical value, above which the bulk property can be obtained, was systematically investigated utilizing a wide range of D/d values (0.5–30) for specimens with a grain size ranging from 0.3 to 2 μm. It was found that the critical D/d value decreases with increasing grain size. A simple analytical model was developed, which is applicable to both micropillar and tensile tests. Comparison of the model equations with experimental data showed that the reduction in yield stress with sample size can be associated with the weaker near-surface zone, which has a reduced strength compared to the specimen interior zone. Furthermore, this study suggests that the size of the near-surface zone may be related to the dislocation structures in near-surface grains, and therefore the overall behavior is governed by the grain size and stacking fault energy.

1. Introduction

It is a well-established fact that the measured mechanical properties of a specific material can change substantially when the specimen dimensions are reduced with respect to the microstructure. These size effects on the mechanical properties have been an important concern in materials science and applied mechanics, and have been first documented by micro-hardness testing by Schulz and Hanemann [1]. These size effects are often categorized as 'intrinsic' or 'extrinsic' [2]. Intrinsic size effects mainly arise from microstructural constraints, such as the grain size or precipitates. The well-known Hall–Petch [3] or Orowan [4] relations have been utilized to predict a material's properties; the strength of a material increases when decreasing the size of these microstructural constraints. The extrinsic size effects are primarily due to dimensional constraints and/or increased effects of the free surface of a specimen. Recent developments in nano/micro-mechanical test systems [5] have enabled active investigations on the specimen size effects, leading to the so-called smaller is stronger phenomenon, which was observed for various single-crystal materials [6].

The interaction of the intrinsic and extrinsic size effects is of concern for many fundamental and commercial interests. Evaluating mechanical properties from reduced-size specimens of polycrystalline has been a major interest for nuclear materials research in recent years [7–9]. This is due to the fact that these tests enable the generation of mechanical data from small volumes, which is especially important for neutron irradiated samples where the radiation dose rate scales with the volume of the material handled [10–14]. Other advantages utilizing reduced-size specimens come from the limited space in research reactors for sample exposure as well as the limited beam penetration depth for the ion-irradiated specimen [15–18]. Miniaturization of the components in electronics, medical or mechanical device production also requires constitutive relations for predicting the materials' behavior during microforming [19]. Therefore, understanding the microstructural and specimen geometry interactions are essential for utilizing these techniques for a large variety of engineering applications.

The effects of the specimen size on the mechanical properties of polycrystalline materials have been studied for decades [20–25]. Specimen size effects on the mechanical properties of various materials have been reported for flat tensile specimens [10,11,20,21]. The measured yield stresses were found to decrease

* Corresponding author. Tel.: +82 31 330 6468; fax: +82 31 330 6469
** Corresponding author. Tel.: +1 510 643 3288; fax: +1 510 643 9685.
E-mail addresses: c.shin@mju.ac.kr (C. Shin), peterh@berkeley.edu (P. Hosemann).

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with decreasing specimen thickness (t) when the value of the thickness divided by the material's grain size (t/d) becomes smaller than a specific critical value. Note that t/d reflects approximately the number of grains across the thickness.

Recently, the size effect on the strength has been investigated for different specimen geometries and/or nanocrystalline materials. Circular compressive nanopillars and square tensile nanopillars were tested for 60 nm grained Ni–4.4%W [23]. Clear size-induced weakening was observed when the value of the specimen's diameter divided by the grain size (D/d) was lower than a critical value. 12 nm-grained Pt nanopillars were compressed, and a similar size weakening was observed [24].

Yang et al. [25] studied the size effect using tensile tests on Cu wires with grain sizes ranging between 2.5 and 36 μm. A clear weakening with decreasing wire diameter was observed, but the critical D/d could not be determined because the D/d values of the tested specimen had a limited range. A simple analytical model was also proposed in [25] to account for the weakening phenomenon in a cylindrical geometry. Numerical or analytical models for a flat tensile geometry can be found in the literature [20,26–29].

From a practical viewpoint, the value of the critical size is of primary interest in determining the specimen size for obtaining bulk mechanical properties. The accumulated experimental results for size-induced weakening suggest that critical values (t/d or D/d for flat plate or nanopillar/wire, respectively) of a material depend on the grain size, specimen geometry and stacking fault energy. In other words, critical values for weakening are a complex function of intrinsic (microstructure, stacking fault energy) and extrinsic (specimen geometry) effects. While the dependence of the critical values on the intrinsic or extrinsic parameter of a specimen is evident, systematic studies to date are limited. Moreover, a few studies have reported an inverse size effect, i.e., size-induced strengthening. One of these is for tensile-tested Ag wire with a grain size ranging from 3.5 to 40 μm [30]. In this case the strength was found to increase with decreasing D/d when D/d is below 3. Another example of this inverse size effect is for 30 nm grain-sized Ni pillars, which shows that the strength of $D/d \sim 5$ is stronger than $D/d \sim 9$ [31]. Hence the coupled effects of grain size and specimen size are much less known.

In this study, size effects on the yield stresses are investigated for microcompression tests of a bulk austenitic alloy with a pillar diameter-to-grain size ratio (D/d) in a wide range of 0.5–30. The effects of grain size on the critical size for weakening are systematically evaluated. A model originally proposed by Yang et al. [25] and also applied for nanopillar tests in [24] is elaborated and extended to a flat tensile geometry. Comparing the experimental results obtained here with the extended model from Yang et al. [25] helps to explain the physical meaning of the model parameters in this work.

2. Experimental

The material used in this study is an austenitic alloy with a chemical composition of Fe–0.0013C–8.14Ni–10.3Cr–7.41Mn in wt%. This type of alloy shows metastable austenite at room temperature after solution treatment at 1100 °C for 12 h. The metastable austenite (γ) transforms into martensite (α') by subsequent cold rolling. Annealing of the cold-rolled specimens transforms the strain-induced α' back into γ. The grain size of the reversed transformed austenite can be controlled by modifying the thermo-mechanical process (cold rolling and annealing) and especially adjusting the annealing temperature. Nano-sized austenite grains can be obtained using this strain-induced transformation ($\gamma \rightarrow \alpha'$) and reverse transformation ($\alpha' \rightarrow \gamma$), which is called SIMRT (strain-induced martensite and reverse transformation) [32]. In this study, the specimens of the alloy, which were cold-rolled to 75% thickness reduction, were annealed at 663, 615, and 575 °C, and the grain sizes of the specimens were 2 ± 0.7, 0.6 ± 0.1, and 0.3 ± 0.1 μm, respectively. The details of the specimen preparation and the grain size measurement can be found in [33].

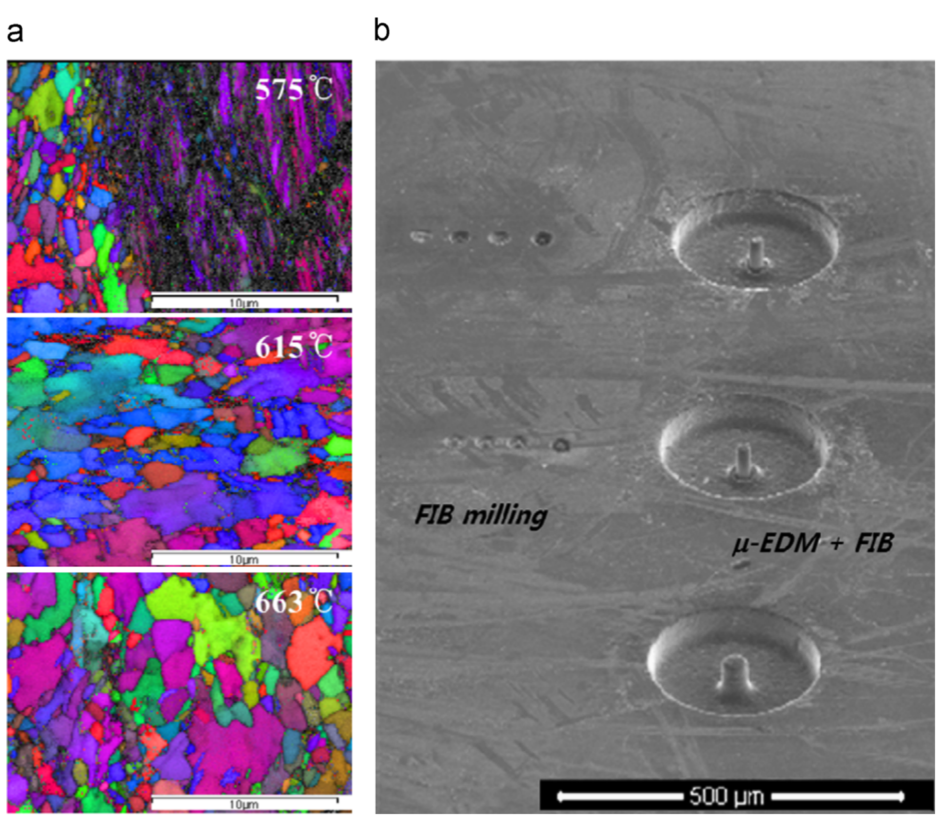

Fig. 1. (a) Orientation maps of each specimen measured using EBSD mapping and (b) SEM image of micropillars fabricated on the surface of the 663 °C specimen.