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Specimen Size Effects on Mechanical Properties of Small-scale Mechanical Testing

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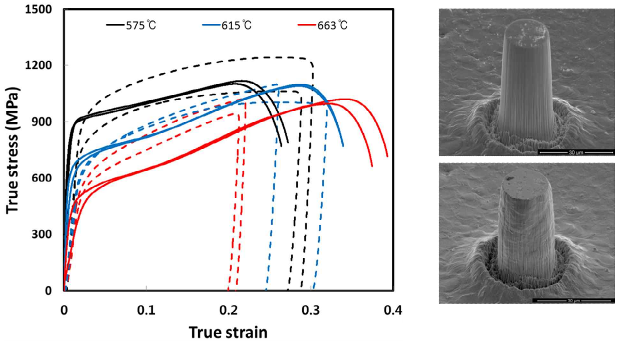
1. Introduction

Effects of specimen size on mechanical properties of polycrystalline materials have been studied for decades [1-3]. Specimen size effects on mechanical properties of various materials have been reported for flat tensile specimens [4-5], and circular compressive nanopillars and square tensile nanopillars [6]. The stresses were found to decrease with decreasing specimen size when the value of the specimen size (s) divided by material's grain size (d) becomes smaller than a critical value. Note that s/d is approximately the number of grains across the specimen size. In practical viewpoint, the value of the critical size is of primary interest in order to determine the specimen size for obtaining bulk mechanical properties. The accumulated experimental results for size-induced weakening suggest that critical values of a material depend on grain size, specimen geometry and stacking fault energy. In other words, critical values for weakening is 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 have been limited. In this study, size effects on yield stresses are investigated for microcompression tests of a metastable bulk austenitic alloy with the pillar diameter to grain size ratio in a wide range from 0.5 to 30. The effects of grain size and lattice defects on the critical size for weakening are systematically evaluated.

2. Methods and Results

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 alloys shows metastable austenite at room temperature after solution treatment at 1100℃ and hot rolling. The metastable austenite transforms into martensite by cold rolling, and subsequent annealing transforms martensite into austenite. 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. In this study, the specimens of the alloy was annealed at 663, 615, and 575℃ and the average grain sizes of the specimens were 2, 0.6 and 0.3 μm, respectively. Circular micropillars with a diameter ranging from 0.6 to 18 μm were fabricated by using focused ion beam (FIB) milling method. Microcompression of the fabricated micropillars was performed using a nanoindenter (NHT2, CSM instruments) equipped with a conical diamond flat punch indenter (45^o cone angle, 20 μm diameter flat end). In order to evaluate the tensile flow curves of each specimen, tensile tests were performed at room temperature with miniaturized flat tensile specimen. The sizes of the gauge section are 1.2 (width in mm) × 5.4 (length in mm) × 0.5 (thickness in mm). All tests were performed at a strain rate 6.7×10^{-4} s^{-1}. The strain was measured using a non-contact extensometer with laser light-sources (laserXtens HP, Zwick).

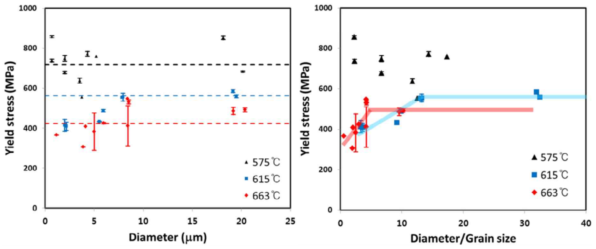
Fig. 1(a) shows the true stress-strain curves of each specimen measured by the tensile tests (solid lines) and the microcompression tests of 18-μm diameter micropillars (dashed lines). True stress-strains were obtained from microcompression tests using the method in ref. [27]. The flow curves of the micropillars with a diameter of 18 μm are comparable to that of the bulk materials. Fig. 1(b) shows SEM images of 18-μm micropillars of the 663℃ specimen before and after compression. Slip traces can be seen on the surface of the deformed micropillar, and no strain localization was found.



(a) (b)

Fig. 1. (a) True stress-strain curves measured by miniaturized tensile tests (solid lines) and micropillar tests (dashed lines) for each specimen (Black: 575℃, Blue: 615℃, Red: 663℃ specimen), (b) Pre- and post-deformed 18-μm micropillars of 663℃ specimen

Fig. 2(a) shows the 0.2% offset yield stresses plotted against pillar diameter for each specimen which has a different grain size. The yield stresses evaluated from the tensile tests are plotted as dashed lines for each specimen. The yield stress decreases with decreasing pillar diameter except for 575℃ specimen. The weakening, i.e., the decrease in yield stresses with decreasing grain size, can be identified more clearly in Fig. 2(b), where the diameter divided by grain size (D/d) is used for the abscissa instead of the diameter. The diameter divided by grain size denotes the average number of grains spanning the diameter of micropillar. Lines are included for visual aid. The weakening has occurred for both 615 and 663℃ specimens, but the values of D/d where the weakening begins are different for each specimen. Larger grain size, i.e. 663℃ specimen, showed smaller D/d value. Note that the yield stresses of the 575℃ specimen showed no size effect and scattered more.



(a) (b)

Fig. 2. 0.2% offset yield stresses plotted against (a) pillar diameter, (b) pillar diameter divided by grain size for each specimen Dashed lines in (a) represent the bulk yield stresses measured by miniaturize tensile tests. Lines are added for visual aid in (b)

The austenitic alloy used in this study was irradiated with 1.5 MeV protons at 300℃. A uniformly damaged layer of 1 dpa (displacement per atom) could be obtained within the depth of 10 μm from the free surface. Micropillars with diameters ranging from 0.6 to 10 μm were fabricated perpendicular to irradiation direction in the damaged layer. Voids were formed, and the size is about 20 nm near the stopping depth (~12 μm) of the ion beam. Much smaller voids were found to be uniformly distributed in the depth where the micropillars were fabricated. The yields strengths of the irradiated materials are shown in Fig. 3. Compared to the unirradiated cases, the weakening effect is decreased and the critical D/d values are decreased due to irradiation.

3. Conclusions

In this study, the specimen size effects on the yield stress are systematically investigated for a bulk metastable austenitic alloy with grain sizes of 0.3, 0.6, and 2 μm. Microcompression tests have been performed on micropillars with a diameter ranging from 0.6 to 18 μm, which enables a wide range of the specimen to grain size ratio (D/d) of 0.5~30 to be studied. The yield stresses were found to decrease with decreasing specimen size, especially when D/d is below a critical value. The critical value was decreasing function of grain size and the density of lattice defects. Care should be taken when determining micropillar size to evaluate bulk mechanical properties for nanocrystalline materials, since the critical D/d value is increasing with decreasing grain size and the weakening is more pronounced in micropillar than flat tensile specimen geometry.

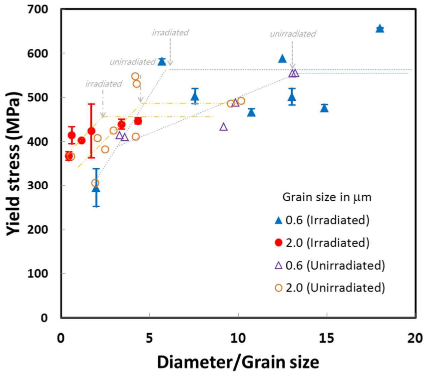


Fig. 3 Yield stresses of the irradiated (filled symbols) and unirradiated (open symbols) specimens plotted against D/d

REFERENCES

[1] N. Igata, K. Miyahara, K. Ohno, T. Uda, Proton Irradiation Creep of Thin Foil Specimens of Type 304 Austenitic Stainless Steel and the Thickness Effects on Their Mechanical Properties, Journal of Nuclear Materials, Vol.122&123, p. 354, 1984.

[2] K. Miyahara, C. Tada, T. Uda, N. Igata, The Effects of Grain and Specimen Sizes on Mechanical Properties of Type 316 Austenitic Stainless Steel, Journal of Nuclear Materials, Vol. 133&134, p.506, 1985.

[3] A. Kohyama, H. Matsui, K. Abe, K. Hamada, K. Asano, Specimen Size Effects on Mechanical Properties of 14MeV Neutron Irradiated Metals, Journal of Nuclear Materials, Vol.155-157, p.1354, 1988.

[4] A. Kohyama, K. Hamada, H. Matsui, Specimen Size Effects on Tensile Properties of Neutron-irradiated Steels, Journal of Nuclear Materials, Vol. 179-181, p.417, 1991.

[5] S. Miyazaki, K. Shibata, H. Fujita, Effect of Specimen Thickness on Mechanical Properties of Polycrystalline Aggregates with Various Grain Sizes, Acta Metallurgica, Vol. 27, p. 855, 1979.

[6] D. Jang, J. R. Greer, Size-induced Weakening and Grain Boundary-assisted Deformation in 60 nm Grained Ni Nanopillars, Scripta Materialia, Vol. 64, p. 77, 2011.