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Evaluation of Mechanical Properties of Oxide Dispersion Strengthened Steel using Micron-sized specimens

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1. Introduction

Material properties are gradually changing as the radiation damage accumulates under the influence of neutron irradiation [1]. The degradation of the mechanical property is one of the key issues determining the safety and performance of structural nuclear materials. Evaluation of the mechanical property changes is a requisite to ensure the reliability of materials used in currently operating nuclear systems and to screen candidate materials for advanced reactor systems. The prime way to evaluate the irradiation effects is to irradiate materials and perform mechanical tests under the same conditions under which the materials are expected to be in service. However, mechanical tests of bulk neutron-irradiated specimens are time- and cost-intensive tasks because the handling of the highly radioactive materials caused by neutron irradiation is difficult and requires high expenses both in time and money.

Small-volume specimens are advantageous in studying the irradiation effects because radioactivity can be minimized by reducing sample size. The cost in the handling of radioactive materials can be lowered as a result. Recently micropillar compression tests have been developed and applied to various materials. In micropillar compression tests, pillars with micron to submicron sizes are uniaxially compressed using a flat punch tip [2]. However the question remains: how accurately can small specimens provide the mechanical properties of a material?

Another advantage of using micropillars is to use in evaluating the properties of an ion irradiated surface layer. Ion irradiation was developed for the purpose of emulating the effect of neutrons without activating materials. Ion irradiation provides flexibility in varying irradiation conditions such as dose, dose rate, and radiation temperature. Hence, there are significant advantages to use ion irradiation in studying the underlying physical mechanisms of radiation damage. However, ion irradiation provides only a small-irradiated volume in the micrometer ranges owing to the limited penetration depth of ions. Nanoindentation tests have been widely used to extract the mechanical properties from such a shallow ion irradiated surface layer [3]. Although the experiment is easy to apply, the interpretation of the experimental data is difficult because of the complicated geometries involved in both ion-irradiated damage profile and indentation, i.e., complex stress state and plastic zone under the indenter. A much simpler stress state without stress gradients can be generated in micropillar compression tests compared to nanoindentation. A micropillar test can be a promising technique for application in studying the irradiation effects on nuclear materials by overcoming the main limitation of both the neutron and ion irradiation mentioned above.

In the present study, we are attempting to apply micropillar tests to characterize the mechanical properties of oxide dispersion strengthened (ODS) steel. We will investigate at what size the yield strengths of ODS steels evaluated by the micropillar tests are comparable with those measured by macroscopic tensile tests. It is expected to use micropillar compression tests combined with ion irradiation once the sample size effects on mechanical properties are revealed.

2. Methods and Results

2.1 Fabrication of ODS micropillars

High-Cr ODS ferritic steel was used in this study. Nominal compositions are Fe-15Cr-2W-0.2Ti-0.35Y_2O_3. Micropillars were made on the surface of ODS plate by using focused ion beam (FIB) system (FEI Nova 2000). The diameter of pillars was ranging from 1 to 5 um. Fig. 1 shows SEM images of ODS micropillars before compression. The aspect ratio (height/diameter) was maintained at between 2 to 4. Pillars were slightly tapered and the taper angle was 2 to 3.5^o.

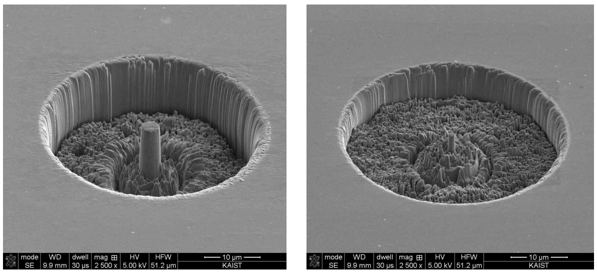
(a) (b)

Fig. 1. Representative ODS micropillars fabricated by FIB with a diameter of 3 μm (a) and 1 μm (b)

2.2 Nanoindentation

A nanoindenter (NHT^2, CSM instruments) was purchased and installed at KAERI. The CSM Indentation Testers are high precision instruments, and can be used for the determination of mechanical properties of almost any type of material. A unique surface referencing technique enables rapid measurement cycle time, negligible thermal drift and negligible system frame compliance. The nanoindenter is equipped with a flat-ended conical diamond punch. The nominal diameter of the punch was 20 μm. The locations of micropillars were identified by high magnification optical microscopy. The pillars were compressed with a load rate of 0.6~6mN/s to induce approximately constant displacement rate for different size pillars. Fig. 2 shows the nanoindenter used in this study.

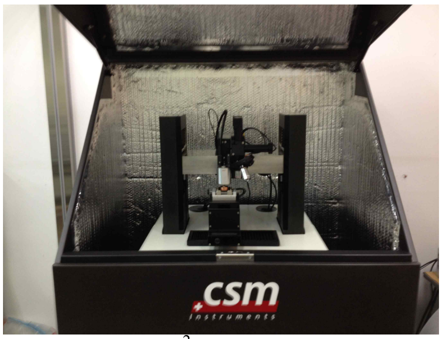

Fig. 2. Nanoindenter (NHT^2, CSM instruments)

2.3 Microcompression tests

The measured load (P) and displacement (d) curves were converted into stress-strain curves. Prior to computing strains, the measured displacement was corrected by subtracting the penetration of the pillar into the underlying substrate and the load-frame compliance of the machine [4]. True stress-strain curves were determined using the conventional constant volume, homogeneous deformation model [5].

Fig. 3 shows representative true stress-strain curves for ODS steels. The measured compressive elastic moduli were consistent with the value (~156 GPa) reported in the literature [6]. The correctly measured elastic modulus indirectly shows that the system is well aligned. We used tilting table to align pillars with the surface of flat punch.

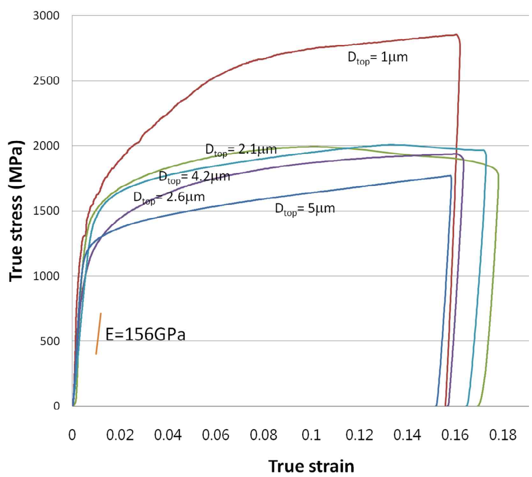

Fig. 3. True stress-strain curves of ODS micropillars

No apparent size effects were identified in micropillars of ODS steels (Fig.4). The yield strengths were evaluated to be ~1.2GPa, which is consistent with the results evaluated by miniaturized tensile tests [7].

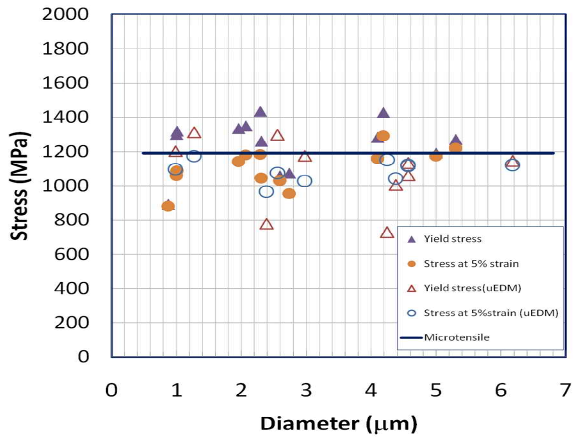

Fig. 4. Size effect on the strength of ODS steel

3. Conclusions

We evaluated the mechanical properties of ODS steels by micropillar compression test. It is found that the bulk mechanical properties can be evaluated by using a micropillar with a diameter as small as 2 μm. The mechanical properties of an irradiated material can be evaluated with a micrometer sized sample. Promising implications of micropillar tests in nuclear applications are evident from this study.

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