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INSTRUMENTED INDENTATION TEST OF TITANIUM ALLOYS AT ELEVATED TEMPERATURE

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Abstract:
The studies of instrumented indentation test at elevated temperature have been increasing in recent years. However, there are few studies about the validity of instrumented indentation test at elevated temperature. For investigating the validation of instrumented indentation test at elevated temperature, we compared the results from the instrumented indentation test with those from micro-bending test, with respect to the heat resistant titanium alloy.

Keywords: instrumented indentation test; elevated temperature; titanium alloys; micro-bending test

1. INTRODUCTION

The studies of instrumented indentation test at elevated temperature have been increasing in recent years. However, there are few studies about the validity of instrumented indentation test at elevated temperature. One of the methods to verify the validity of instrumented indentation test at elevated temperature is comparing with another mechanical tests. Micro-bending test is useful method for investigating the effects of surface change by elevated temperature and the reaction between indenter and measured materials on the results of instrumented indentation test. It is because the test can be operated in the same environment as instrumented indentation test, and the mechanical properties is evaluated by bending the cantilever regardless of the surface change.

In this study, for investigating the validation of instrumented indentation test at elevated temperature, we compared the results from the instrumented indentation test with those from micro-bending test, with respect to the heat resistant titanium alloy, IMI 834. Although the maximum operating temperature of this alloy is 590 °C [1], the mechanical properties at elevated temperature are gradually changed even under 590 °C [2]. A part of the experimental data in this paper is based on our previous study about titanium alloys [3].

2. METHOD

The sample was cut out IMI 834 (Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si). Nanoindentation tests and micro-cantilever bending tests were carried out by using a commercial nanoindentation tester (Hysitron, TI950 Tribo-Indenter) with a Berkovich indenter at 25 °C and 350 °C. In nanoindentation test, the maximum load was 5000 μN. Elastic modulus and hardness values were calculated from unloading part of load-displacement curve [4]. For verifying the damage of the diamond indenter, fused silica as the reference material was measured by indentation tests before and after measuring the titanium alloys at elevated temperature. The maximum load for fused silica was 8000 μN, to cover the indentation depth of titanium alloys.

For micro-cantilever bending tests, we fabricated micro-cantilevers of IMI 834 using focused ion beam (FIB) milling system. Figure 1 shows the micro-cantilever fabricated by FIB. The cantilevers have an equilateral triangular cross-section. The length and width of the cantilevers were 40 μm and 10 μm, respectively. The load was applied at 30 μm from the base of the cantilever.

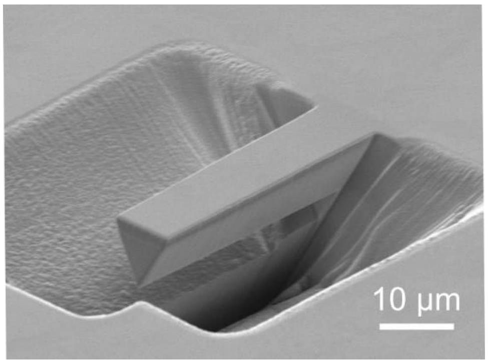

Figure 1: The micro-cantilever fabricated by FIB

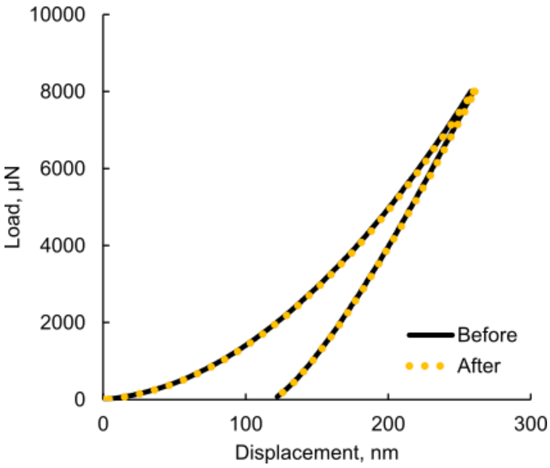

Figure 2: The comparison of load-displacement curve of the fused silica between 1st and 2nd nanoindentation tests

The elastic modulus was calculated from the linear fitting of the stress-strain curve in the region where the strain was 0.001–0.015. The 0.2 % proof stress was calculated from the intersection point of the stress-strain curve and the line shifted by 0.002 from the linear fitting line in the elastic region.

A series of experiments were carried out in the following order.

1. 1st nanoindentation test for fused silica.
2. Nanoindentation and bending tests for IMI 834 at 25 °C.
3. Nanoindentation and bending tests for IMI 834 at 350 °C.
4. 2nd nanoindentation test for fused silica.

Titanium has hcp-structure, which has anisotropy; thus, the hardness and elastic modulus depend on the c-axis inclination angle [5], [6]. The electron backscattering pattern (EBSD) measurements were conducted to obtain crystal structure information. For focusing on the temperature dependence, we show the results of tests, which deformed grains whose c-axis was limited to within 60-80° (at indented position in indentation test and base part of the cantilever in bending test).

3. RESULTS AND DISCUSSION

Figure 2 shows the load-displacement curve of the fused silica of 1st and 2nd nanoindentation tests. The maximum depth of 1st and 2nd measurement of fused silica was 259.7 nm and 261.1 nm, respectively. In addition, the elastic modulus of fused silica of 1st and 2nd measurement was 71.3 GPa and 69.9 GPa, respectively. The hardness values of fused silica of 1st and 2nd measurement was 9.32 GPa and 9.33 GPa, respectively. These results suggest that the diamond indenter was not damaged through measurements of the titanium alloy at 350 °C.

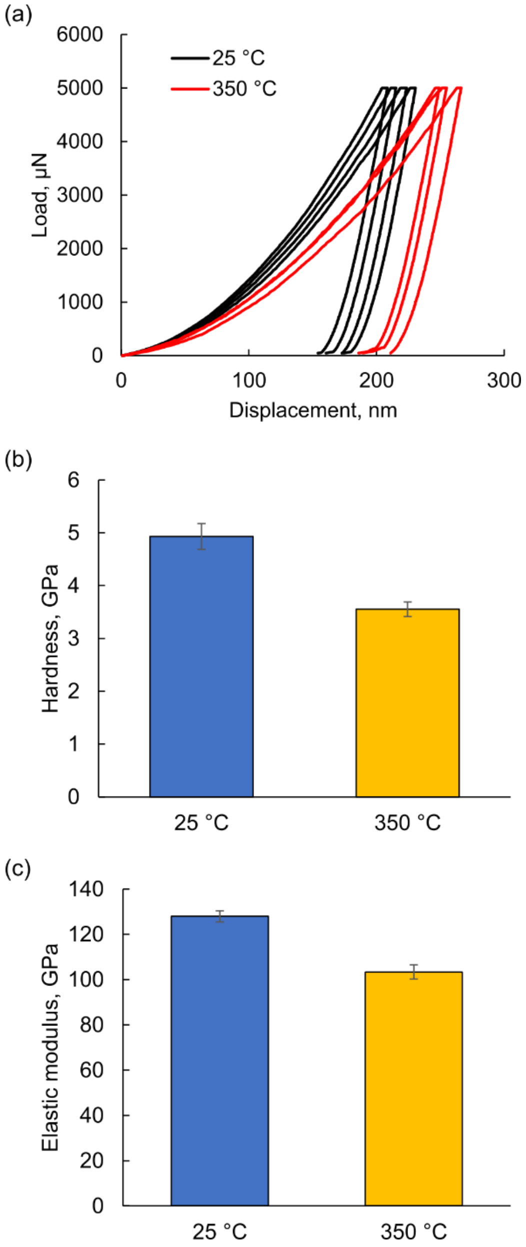

Figure 3: Results of nanoindentation tests with respect to IMI 834 at 25 °C and 350 °C. (a) Load-displacement curves. (b) Mean hardness. (c) Mean elastic modulus

Table 1: Elastic modulus and hardness by nanoindentation tests. Values reported as mean ± standard deviation.

	Elastic modulus, GPa	**Hardness, GPa**
25 °C	127.9 ± 2.5	4.93 ± 0.24
350 °C	103.3 ± 3.1	3.55 ± 0.14

Figure 3a shows the load-displacement curve of IMI 834 by nanoindentation test at 25 °C and 350 °C. The indentation depth at 350 °C was larger than that at 25 °C. Figure 3b-c and Table 1 show mean hardness and elastic modulus. The hardness and elastic modulus at 350 °C were smaller than those at 25 °C. The difference between 25 °C and 350 °C was about 30 % in hardness, and the difference was about 20 % in elastic modulus.

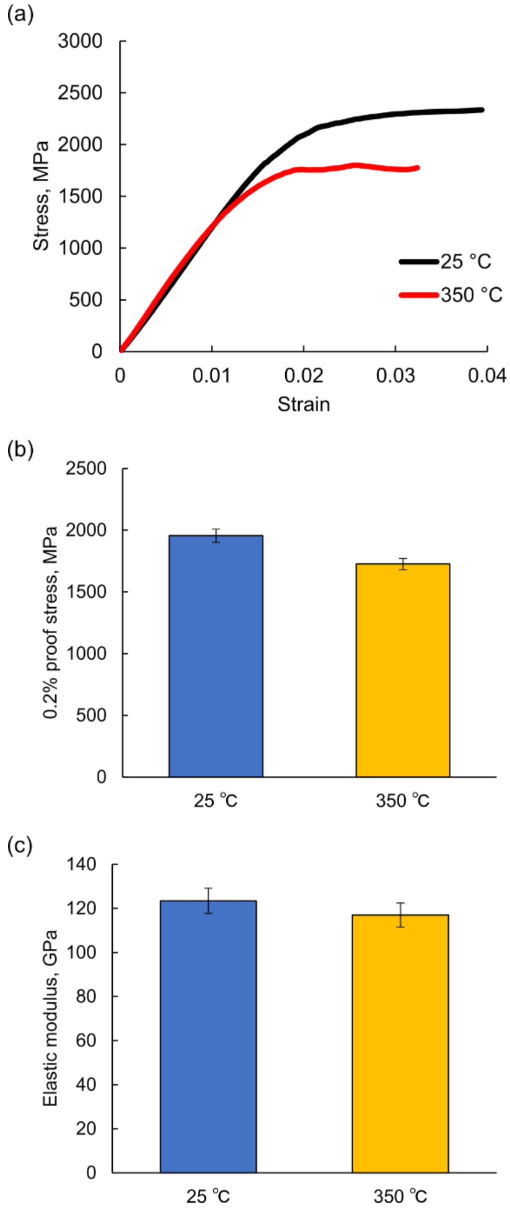

Figure 4: Results of micro-cantilever bending tests with respect to IMI 834 at 25 °C and 350 °C. (a) Example of stress-strain curves obtained from micro-bending tests at 25 °C and 350 °C. (b) Mean 0.2% proof stress. (c) Mean elastic modulus

Figure 4a shows the example of the stress-strain curves obtained from micro-bending tests at 25 °C and 350 °C. Yield stress at 350 °C is lower than that at 25 °C. Figure 4b-c and Table 2 show the mean elastic modulus and mean 0.2 % proof stress of three micro-cantilevers in each of 25 °C and 350 °C. The 0.2 % proof stress at 25 °C and 350 °C was 1956 MPa and 1726 MPa, respectively. The elastic modulus obtained from the elastic region of stress-strain curve at 25 °C and 350 °C was 123.4 GPa and 117.0 GPa. The difference between 25 °C and 350 °C was about 12 % in the 0.2 % proof stress, the difference was about 5 % in elastic modulus.

Focusing on the plastic deformation, the difference in hardness between 25 °C and 350 °C was 30 % in nanoindentation test, while the difference in 0.2 % proof stress was 12 % in micro-bending test. The resistance to the plastic deformation of 350 °C was lower than that of 25 °C in both nanoindentation tests and micro-cantilever bending tests. This result suggests that the nanoindentation test can obtain mechanical properties about plastic deformation even at elevated temperature, consistent with other testing. In addition, the difference in elastic modulus between 25 °C and 350 °C in nanoindentation test is about 20 %, while the difference in bending test is about 5 %. The elastic modulus of 350 °C was lower than that of 25 °C in both nanoindentation tests and micro-cantilever bending tests, although the difference is different between nanoindentation and bending tests.

Table 2: Elastic modulus and 0.2% proof stress by micro-bending tests. Values reported as mean ± standard deviation.

	Elastic modulus, GPa	**0.2 % proof stress, MPa**
25 °C	123.4 ± 5.7	1956 ± 6
350 °C	117.0 ± 5.4	1726 ± 5

When the nanoindentation test is performed at elevated temperature, it is important to consider the oxidation of the sample surface [7], [8]. Titanium alloy is known to be oxidized by high temperature. The previous study indicated that the relationship between titanium oxidation and hardness was evaluated in the pure titanium and Ti-0.5Al-0.45Si-0.2Nb alloy [9]. When the titanium was oxidized for long time (exposed in the air at 800 °C for 100 h), micro-Vickers hardness increased near the surface. Therefore, oxidation of the titanium increase the hardness. However, the hardness at 25 °C was higher than that at 350 °C, in which the titanium oxide is assumed to be easily generated, in this study. IMI 834 is well-designed high-tempearture alloy, and have high yield strength, high heat resistance and high oxidation resistance, compareing with Ti-0.5Al-0.45Si-0.2Nb alloy [9]. In addition, the mechanical tests in this study were performed for short time (1~2 h) and in intermediate temperature (350 °C). Oxidation of IMI 834 seemed to be low in this study, because the oxidation of IMI 834 increases when the temperature is over 600 °C and the oxidation time is long [10]. Besides, the result of plastic property in nanoindentation test (approximately 200 nm scale) showed a similar tendency to that in micro-bending test (approximately 10 µm scale) which was little affected by oxidation. From these reasons, the oxidation layer was assumed to be very thin, and there are little effects of the oxidation of titanium on the mechanical tests in this study. The result that the hardness and the proof stress at 25 °C were higher than those at 350 °C, indicated the IMI 834 itself easily deformed by incresing tempearture.

In addition, the reaction between the indenter and sample surface was also concerned [7], [8]. As already mentioned, there was not change in the indenter from the result of fused silica before and after high temperature measurement. Moreover, the result of nanoindentation test was similar tendency to the result of micro-bending test. The reaction between the indenter and sample surface significantly affects the nanoindentation test, while the reaction slightly affects the micro-bending test. Considering the result of nanoindentation test was similar tendency to that of micro-bending test, we concluded that there are little effects of the reaction between the indenter and sample surface on the nanoindentation tests in this study.

4. SUMMARY

In this study, for investigating the validation of instrumented indentation test at elevated temperature, we compared the results from the instrumented indentation test with those from micro-bending test, with respect to the heat resistant titanium alloy. The difference in the mechanical properties between 25 °C and 350 °C in instrumented indentation test is similar tendency to the difference in the mechanical properties in micro-bending test. This result suggests that the instrumented indentation test can obtain mechanical properties even at elevated temperature, consistent with other testing.

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