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Evaluation of High-Temperature Tensile Properties of Ti-6Al-4V Using Instrumented Indentation Testing

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Since materials used in or exposed to high-temperature environments can undergo variation or degradation of mechanical properties, it is important to evaluate mechanical properties at high temperature, in particular for structural applications and aerospace materials. Instrumented indentation testing (IIT) is widely used to evaluate such mechanical properties of materials as tensile properties, residual stress, fracture toughness, etc., exploiting theoretical approaches to indentation mechanics. In this study, we used IIT to evaluate variations in tensile properties with temperature of the Ti alloy Ti-6Al-4V, a candidate material for aerospace applications, using a high-temperature chamber and a modified representation method. Comparison of our results with conventional uniaxial tensile test results showed good agreement (within a 10% error range) in yield strength and ultimate tensile strength. This confirms the potential of IIT for evaluating to evaluate high-temperature tensile properties of metallic materials and for research on material behavior in various temperature conditions.

Keywords: indentation, strength, tensile test, Ti-6Al-4V, high temperature

1. INTRODUCTION

Instrumented indentation testing (IIT) (also known as nanoindentation), which measures load and displacement continuously during indentation testing, is widely used to evaluate mechanical properties of materials on multiple scales. Originally developed for measuring the hardness and elastic modulus of thin films in nanoscale [1], IIT has been extended to macroscale to evaluate mechanical characteristics such as tensile properties [2-7], residual stress [8-12], and fracture toughness [13-15] using theoretical approaches to indentation mechanics. As a mechanical testing method, IIT has many advantages: it is simple and nondestructive so that in-field and in-use testing can be performed, multiscale experimental design is possible using a single method, and various properties can be obtained from a single test. Furthermore, IIT can be used for high-temperature testing simply by combining IIT equipment with a high-temperature chamber [16].

Since materials used in or exposed to high-temperature environments can undergo variation or degradation in mechanical properties, it is important to evaluate mechanical properties at high temperature, in particular for structural application or aerospace materials [30]. Tensile properties such as yield strength (YS) and ultimate tensile strength (UTS) obtained by uniaxial tensile testing have been commonly used as high-temperature mechanical properties [17]. Although uniaxial tensile test is a classic mechanical test, it has some disadvantages for high-temperature testing: a specific specimen shape is needed, heating and cooling must be repeated for each test, and controlling complex temperature conditions is difficult. IIT has many advantages as an alternative method for high-temperature testing: any specimen shape can be used, multiple tests on single specimens under the same heating conditions are possible, and various temperature conditions can be controlled as variable in the experimental design.

Much work has focused on theoretical, numerical and experimental modelling for evaluating tensile properties using IIT [2-7], and, as suggested first by Tabor [2], a representation method is widely used for that. In the representation method, representative stress and strain are defined by analyzing the stress field in the indented material beneath a spherical indenter and using derived representative stress-strain points to obtain tensile properties such as YS, UTS, strain hardening exponent by several methods [18-20].

Here we use a uniaxial tensile test and IIT to investigate the variation of tensile properties at high temperatures of Ti-6Al-4V, a Ti alloy widely used in aerospace applications. We performed uniaxial tensile tests and IITs at various temperatures using a high-temperature chamber to evaluate the tensile properties of Ti-6Al-4V. In IIT, we modified the representation method for non-ferrous Ti alloy, since the repre-

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sentation method is generally confirmed for ferrous metals like steels. The experimental results are compared with temperature and shows good agreement in both uniaxial tensile testing and IIT.

2. THEORETICAL MODELING

Recently many researchers have worked to improve the representation method, and as a result the ISO has issued an international standard, ISO/TR 29381, for the method [21]. As described in the technical report, the algorithm for evaluating tensile properties using IIT has four steps: 1) determination of contact area, 2) determination of representative stress and strain, 3) fitting to constitutive Eq., 4) evaluation of tensile properties. The most important step in the algorithm is determination of representative stress and strain. Representative stress is given by

$$\sigma = \frac{P_m}{\psi} = \frac{1}{\psi}\frac{L}{A_c} \tag{1}$$

where P_m is mean pressure, Ψ is plastic constraint factor, L is indentation load, and A_c is contact area at the load.

Tabor [2] and other researchers [22, 23] have suggested a plastic constraint factor for metallic materials of constant value about 3 when indentation behavior is in the fully plastic regime. However, if the indentation process is not in the fully plastic regime, the plastic constraint factor is no longer constant [24]. According to Kim *et al*. [25], the indentation process covers both the elastic-plastic transition regime and fully plastic regime for metallic materials having relatively low elastic modulus and high yield strength like our Ti alloy, as shown in Fig. 1. Consequently, we modified the plastic constraint factor to evaluate tensile properties of the Ti alloy using the representation method.

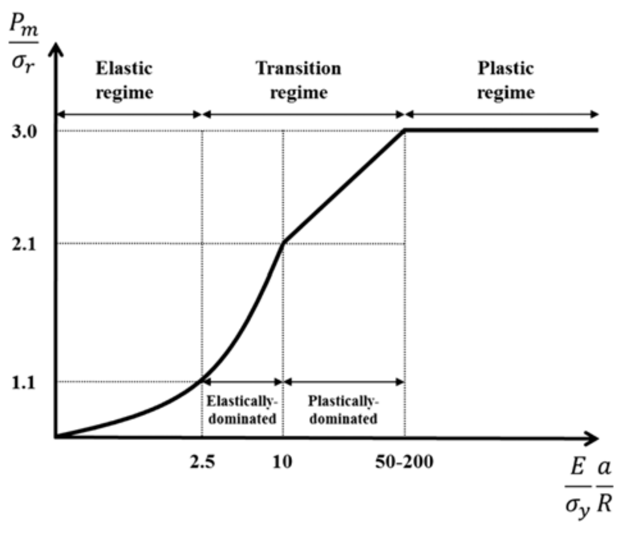

Fig. 1. Schematic representation of relationship between plastic constraint factor and indentation parameters from Park and Pharr [32].

Furthermore, it is expected that this behavior can be changed by variation of temperature and the algorithm may be modified for high-temperature application. Although little work has been done on the effect of temperature on the plastic constraint factor, Kumaraswamy and Venkataraman investigated the effect of temperature on the plastic constraint factor of Ti-6Al-4V under static indentation conditions [33]. According to Kumaraswamy and Venkataraman, the plastic constraint factor of Ti-6AL-4V was independent of temperature as a constant value in the fully plastic regime. In the elastic-plastic transition regime, the plastic constraint factor increased with the normalized indentation strain, $E/\sigma_y*a/R$, and reached the constant value in fully plastic regime at the same $E/\sigma_y*a/R$, which is almost independent of temperature, even though there is some deviation with temperature in the early stages of the elastic-plastic transition regime. These results means that temperature does not influence the constraint effect at least in Ti-6Al-4V and in the temperature range investigated, and the change in deformation behavior with temperature is applied to both mean pressure and the normalized indentation strain. Therefore, in this study we treat the plastic constraint factor and the algorithm at high temperature for Ti-6Al-4V in the same way as at room temperature.

We approximated the plastically dominated transition regime as a linear function given by

$$\psi = 0.016 \times \frac{E}{\sigma_y}\frac{a}{R} + 2.09 \tag{2}$$

from Kim's data and used constant 3 in the fully plastic regime. Another point we considered is determining the boundary between the transition and the fully plastic regime. Kim *et al*. [25] found that, for Ti alloys, when $E/\sigma_y*a/R$ reaches the value of 60, the indentation process is in the fully plastic regime, and we use this value here.

Representative strain is initially defined as $0.2a/R$ in Tabor [2]'s experimental approach, but this shows limited agreement for some steels. Finally, using the geometrical relationship between indentation radius and indentation depth, representative strain is expressed in the mathematical form of tangent functions [18]:

$$\varepsilon = \frac{\propto}{\sqrt{1-(a/R)^2}}\frac{a}{R} = \propto \tan\gamma \tag{3}$$

where α is correlation constants of 0.14 by the finite element method and γ is the contact angle between the indenter and the reference plane of material surface.

With Eqs. (1) and (3), representative stress and strain points are obtained from each unloading point in an indentation load-depth curve. From these stress-strain points, as fitted by a constitutive equation such as the Hollomon equation $\sigma = K\varepsilon^n$, YS and UTS can be derived by 0.2% offset method and the concept of instability in tension [17].

3. EXPERIMENTS

Ti-6Al-4V alloy samples were prepared for uniaxial tensile tests and instrumented indentation tests. For tensile tests, plate-type specimen with gauge length 25 mm and thickness 6 mm were prepared according to the ASTM E21 and E8 standards [26,27]. For indentation tests, since there is no specimen size requirement, rectangular-shaped specimens 5 cm × 5 cm × 2 cm were prepared. Specimen surfaces were mechanically ground with emery paper and finely polished with 1 μm alumina powder. Chemical compositions of specimens are summarized in Table 1.

Uniaxial tensile tests were performed using an INSTRON 5582 (Instron, Inc., USA) with an environmental chamber at cross-head speed 0.5 mm/min. Testing temperatures were varied at 100 °C intervals from room temperature to 500 °C and, for thermal stabilization of the specimen, tests were performed 30 minutes after testing temperature was reached. Tests at each temperature were repeated three times to check repeatability.

Instrumented indentation tests were performed using AIS2100 (Frontics, Inc., Korea) with force resolution 0.06 N and displacement resolution 0.1 μm. A tungsten carbide spherical indenter with radius 250 μm was used, and 15 partial unloading tests were conducted. Maximum indentation depth was 100 μm, the unloading ratio of each unloading was 50%, and the indentation loading/unloading rate was 0.3 mm/min. system, High-temperature indentation tests were performed in the high-temperature chamber under the same conditions as for uniaxial tensile tests.

Table 1. Chemical composition of Ti-6Al-4V

Components	Ti	Al	V	Fe	C
wt%	Bal.	6.75	4.5	0.4	0.1

Fig. 2. High-temperature chamber system with IIT equipment.

Figure 2 shows the high-temperature chamber system for instrumented indentation testing. A vacuum environment was applied to prevent oxidation of specimen and equipment. Specimen, indenter and stage were all heated by halogen lamp and their temperatures were monitored in real time by thermocouples. Specified testing temperature range is from room temperature to 650 °C.

4. RESULTS & DISCUSSION

4.1. Uniaxial tensile test

High-temperature uniaxial tensile test results are shown in Fig. 3. For comparison by temperature, engineering stress-strain curves of Ti-6Al-4V alloy at each temperature are plotted in this one figure. With increasing temperature, stress-strain curves display a downward trend, yield strength is decreased and total elongation is increased.

This behavior can be easily explained: the decrease in strength with increasing temperature is attributed to an increase in mobility of atoms and dislocations in the material due to the influx of thermal energy. If the mobility of atoms and dislocations activates at high temperatures, deformation mechanisms such as dislocation climb become easier than at room temperature, so the strength, defined as resistance to deformation, of the material is reduced and ductility is improved. Detailed tensile properties analyzed from the stress-strain curves of this alloy are given in Table 2. Figure 4 shows a decreasing trend of yield strength and tensile strength with increasing temperature. The degree of decrease is similar in both yield strength and tensile strength.

The change in deformation behavior with temperature can

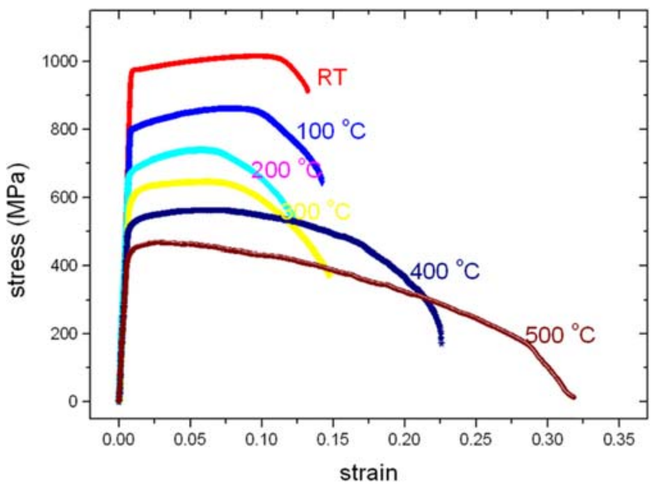

Fig. 3. Engineering stress-strain curves with temperature from uniaxial tensile tests.

Table 2. Tensile properties with temperature from uniaxial tensile test and IIT

Testing temperature (°C)	YS (MPa)			UTS (MPa)		
	Tensile test	IIT	Error (%)	Tensile test	IIT	Error (%)
RT	968.46	948.71	-2.0	1015.50	1044.14	2.8
50 °C	-	900.41	-	-	960.12	-
100 °C	796.71	828.37	4.0	864.48	882.89	2.1
150 °C	-	760.56	-	-	835.67	-
200 °C	667.47	697.07	4.4	739.58	781.47	5.7
250 °C	-	655.78	-	-	730.76	-
300 °C	566.23	601.42	6.2	646.17	672.47	4.1
350 °C	-	552.93	-	-	605.26	-
400 °C	488.36	492.59	0.9	561.87	536.21	-4.6
450 °C	-	461.49	-	-	516.38	-
500 °C	435.98	448.70	2.9	466.88	506.07	8.4

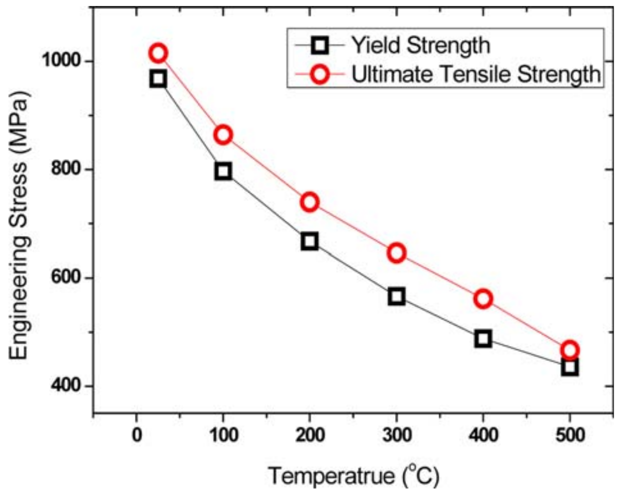

Fig. 4. Variation of tensile properties with temperature from uniaxial tensile tests.

be identified by analyzing the specimen fracture surface. Figure 5 shows photos of tested specimens and identifies the fracture surfaces. For the specimen tested at room temperature, the fracture surface shows brittle-fracture characteristics like a flat cutting surface. For the specimen tested at high temperature, however, the fracture surface shows ductile fracture characteristics such as the cup and cone shape in Fig. 5.

4.2. Instrumented indentation test.

Before discussing high-temperature indentation testing results, we compare the room-temperature indentation test result with the uniaxial tensile test result. As shown in Fig. 6, representative stress-strain points from indentation testing show good agreement with a true stress-strain curve from tensile testing. In addition, as shown in Table 2, tensile properties analyzed from stress-strain data also match well, with error range within 3%. Therefore, it is expected that representative method using instrumented indentation test can be applied in high-temperature testing.

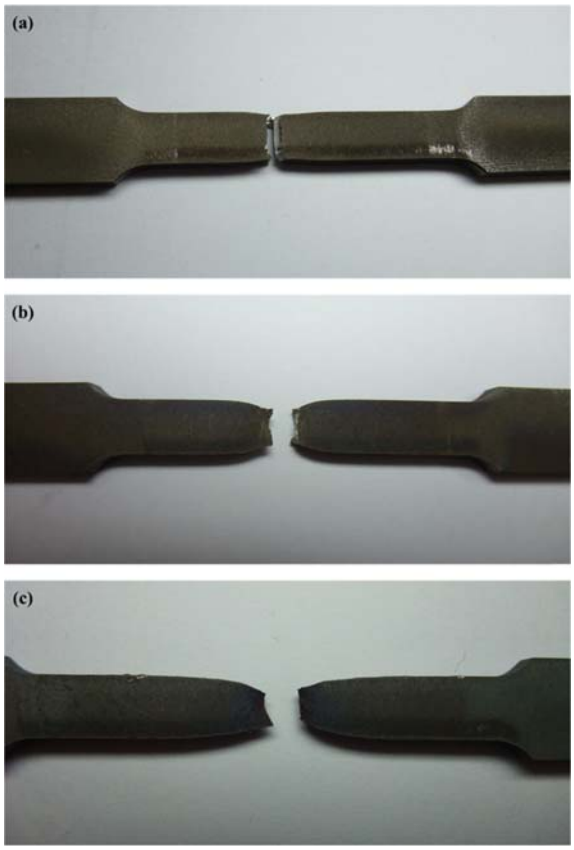

Fig. 5. Fracture surfaces of Ti-6Al-4V specimen tested at (a) RT, (b) 200 °C, and (c) 500 °C

Figure 7 shows indentation load-displacement curves for the Ti alloy at various temperatures. As in the uniaxial tensile test results, indentation curves showed a downward trend with increasing temperature; that is, the indentation load at the same indentation depth decreased with temperature by thermal softening. Table 2 evaluates and details the indentation tensile properties using representation method.

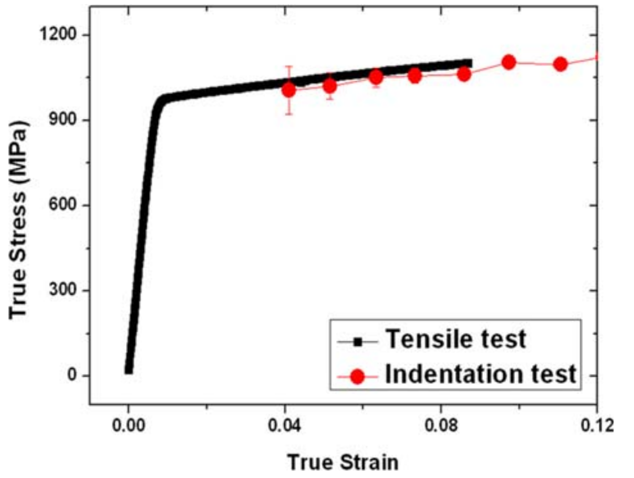

Fig. 6. Comparison of stress-strain curves at room temperature from uniaxial tensile test and IIT.

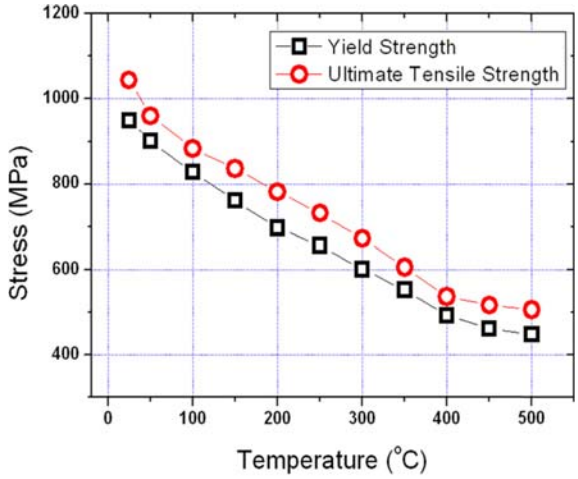

Fig. 8. Variation of tensile properties with temperature from IIT.

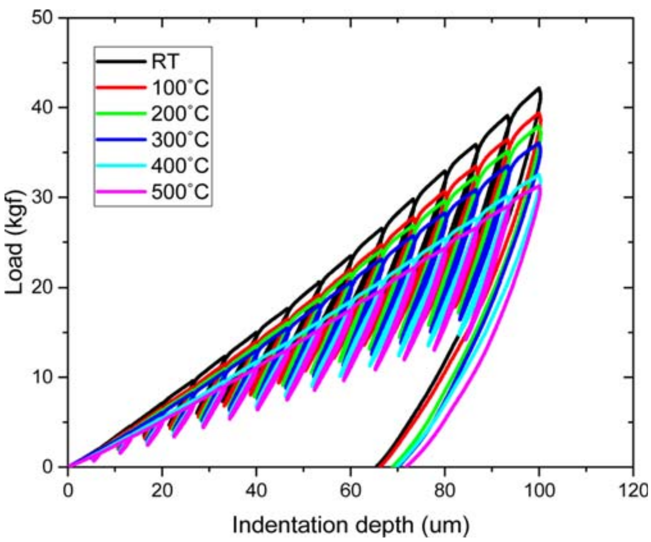

Fig. 7. Indentation load-depth curves with temperature from IIT.

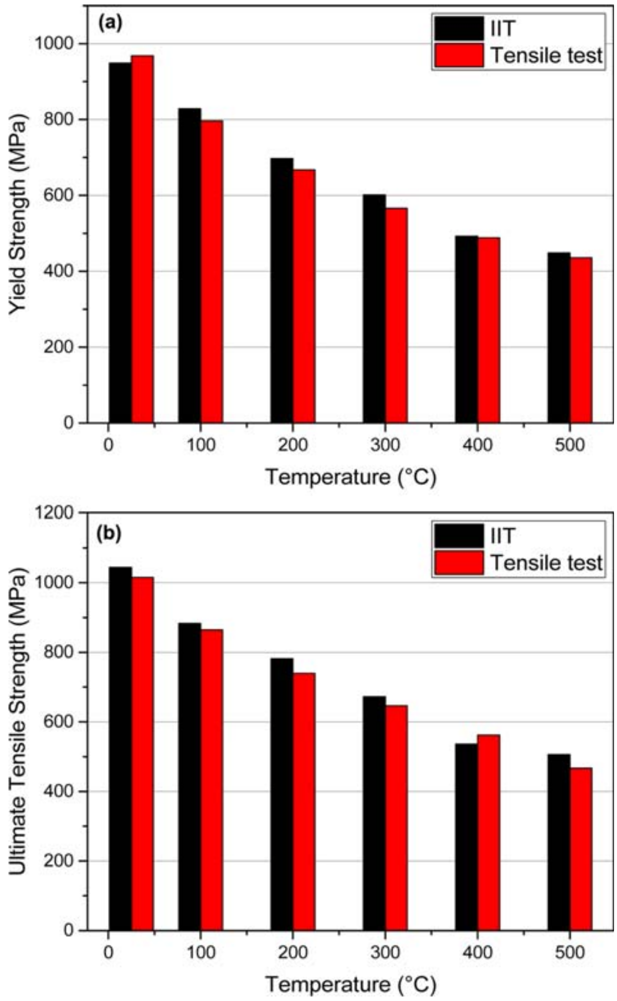

Fig. 9. Comparison of tensile properties with temperature from uniaxial tensile test and IIT. (a) yield strength and (b) ultimate tensile strength.

Figure 8 plots yield strength and tensile strength against temperature. Again, the trends are similar to the uniaxial tensile test results in Figure 4. In the temperature range from RT to 500 °C, since the Ti alloy has no phase transformation or critical change in microstructures [28], it appears that tensile properties steadily decrease with temperature. Figure 9 compares the indentation tensile properties with those from uniaxial tensile tests; again, they are in good agreement within 10% error range.

Comparisons are also made with tensile test results in the literature [29-31]. For comparison with literature values, we converted the tensile property values at high temperatures to percentages of room-temperature values, as shown in Fig. 10. Although yield strength was somewhat overestimated in the 200~300 °C range and tensile strength was somewhat underestimated at 400~500 °C, the values and trends are generally in good agreement with previous research. We conclude that instrumented indentation testing is accurate for evaluating

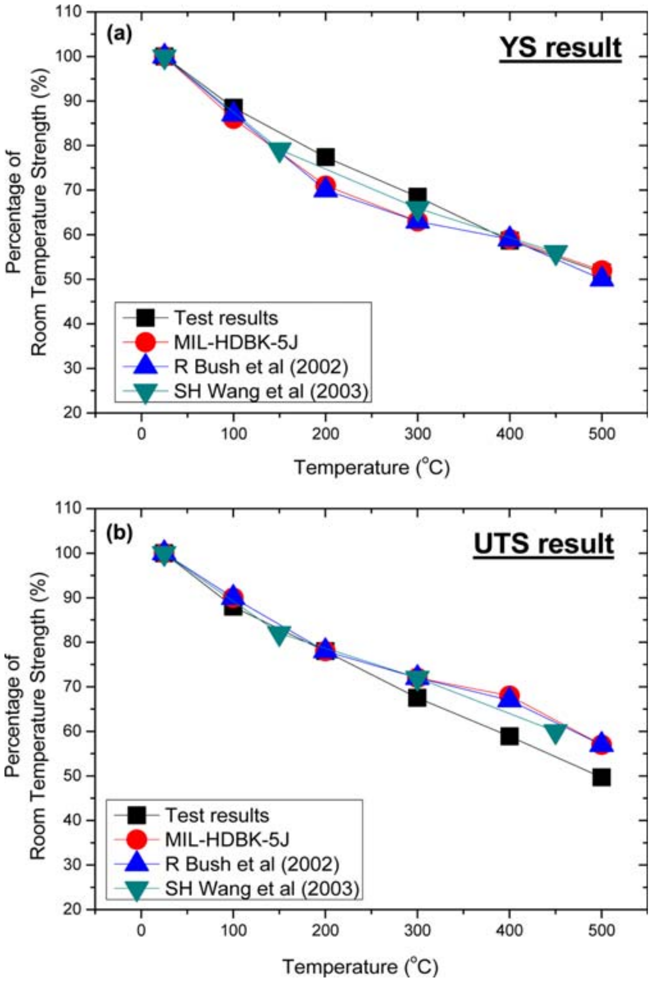

Fig. 10. Comparison of tensile properties with those in the literature. (a) yield strength and (b) ultimate tensile strength.

the high-temperature tensile properties of Ti alloy.

5. CONCLUSIONS

This study evaluated the mechanical properties of the Ti-6Al-4V alloy, a candidate material for aerospace applications exposed to high temperatures. Using uniaxial tensile and instrumented indentation testing, tensile properties such as yield strength and ultimate tensile strength at various temperatures were evaluated and compared. With increasing temperature, the tensile properties of Ti alloy are reduced due to activation of atoms and dislocation mobility. The values of tensile properties and trends with temperature using a high-temperature chamber instrumented indentation system show good agreement with high-temperature tensile test results and also the results in the literature. Due to the advantages of instrumented indentation testing for high-temperature testing, such as its simple and nondestructive nature, brief testing time, no specimen size requirement, capacity to perform multiple tests using one specimen, and easy temperature control, this test can be used as an alternative for evaluation of tensile properties at high temperatures and material behaviors under various temperature conditions such as exposure time control, rapid change in temperature, etc.

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