Microscopic Measurement of the Stress-Strain Relation for Commercially Pure Titanium

Introduction

Because it is easily formed and inexpensive relative to its alloys, pure titanium is the first choice for titanium parts that undergo little external loading[1]. In larger volumes, the strength of titanium is measured by means of a macro-scale tensile or compression test, whereby a sample of uniform cross-section is stretched or compressed uniaxially while continuously monitoring the resulting change in length. The stress is defined as the applied load divided by the cross-sectional area and the strain is defined as the change in length divided by the original length. While the deformation remains elastic, the strain increases in proportion to the stress, with the constant of proportionality being the Young's Modulus of the material, E. The Young's Modulus of pure titanium is 120GPa[2]. The onset of plasticity is identified as the yield point, or the point at which the strain begins to increase disproportionately to the stress. Beyond the yield point, a variety of microstructural mechanisms determine the relation between stress and strain; these include dislocation motion and entanglement, grain boundary sliding, micro-fracturing, etc. Depending on the microstructure, the yield stress for pure titanium may be anywhere in the range of 170 – 480MPa[3].

When utilized in small volumes, pure titanium may be stronger[4] and thus may have new commercial applications. Motivated by the goal of measuring yield strength and hardening at the microscopic scale, nanoindentation is used to measure the stress-strain relation of pure titanium using a flat-ended cylindrical punch with a diameter of only 10µm.

Theory

KLA's patented technique[5] for calculating the stress-strain curve from flat-punch nanoindentation data involves first properly scaling the indentation depth, h, so that it corresponds to the uniaxial strain, ε, and then properly scaling the mean pressure of the contact, pm, so that it corresponds to the uniaxial stress, σ. The strain is defined by scaling the indentation depth as:

$$\varepsilon = \left(\frac{2}{\pi}\right)\left(\frac{h}{a}\right) \qquad \text{Eq. 1}$$

with a being the radius of the flat-ended punch. The stress is defined by scaling the mean pressure as:

$$\sigma = \zeta p_m \qquad \text{Eq. 2}$$

where ζ is not a fixed number, but is calculated based on the degree of plasticity, as quantified by the parameter S*, defined as:

$$S^* = \frac{S_L}{S} \qquad \text{Eq. 3}$$

where S^L is the instantaneous slope of the loading curve, and S is the elastic contact stiffness. It should be noted that in the limit of a completely elastic contact, S* takes the upper limit of unity, because the loading slope is equal to the contact stiffness; in the limit of a completely plastic contact, S* takes the lower limit of 0, because the unloading slope is infinite. With dynamic nanoindentation, ζ is calculated throughout the loading cycle for use in Equation (2) and is defined as:

$$\zeta = 0.39690(S^*) + 0.32180, \qquad \text{Eq. 4}$$

where the constants were determined by hundreds of finite-element simulations on a wide variety of materials as giving the best agreement between the stress-strain curves obtained by the method and the stress-strain curves which were used as input to the simulations.

Experimental Method

The KLA iMicro nanoindenter, having a flat-ended cone with a face diameter of 10µm and an included angle of 90°, was used to perform 12 indentations on a prepared sample of commercially pure titanium using the test method "Flat Punch on Metals for Stress-Strain." The indent spacing was 75µm. For each indentation, the indenter was pressed in to the surface at a constant displacement rate of 50nm/sec to a peak depth of 1600nm. A small oscillation of 2nm at 110Hz was superimposed upon the semi-static loading in order to measure the elastic contact stiffness throughout loading.

The KLA test method "Flat Punch on Metals for Stress-Strain" implements the above theory in a practical way. First, the Young's modulus of the metal is required as an input, either from literature or from prior measurement by standard nanoindentation. The theory for measuring stress and strain is not valid until full contact is achieved between the flat punch and the surface. In the course of the test, the point of full contact is marked when the criteria

$$S > 2Ea \qquad \text{Eq. 5}$$

is met, which is essentially the stiffness relation deduced by Sneddon[6]. Beyond this point, the stress-strain relation is determined according to the Equations 1-4. These stress-strain ordered pairs beyond the point of full contact are fit to the form of power-law hardening

$$\sigma = K\varepsilon^{n} \qquad \text{Eq. 6}$$

in order to obtain best-fit values for K and n.

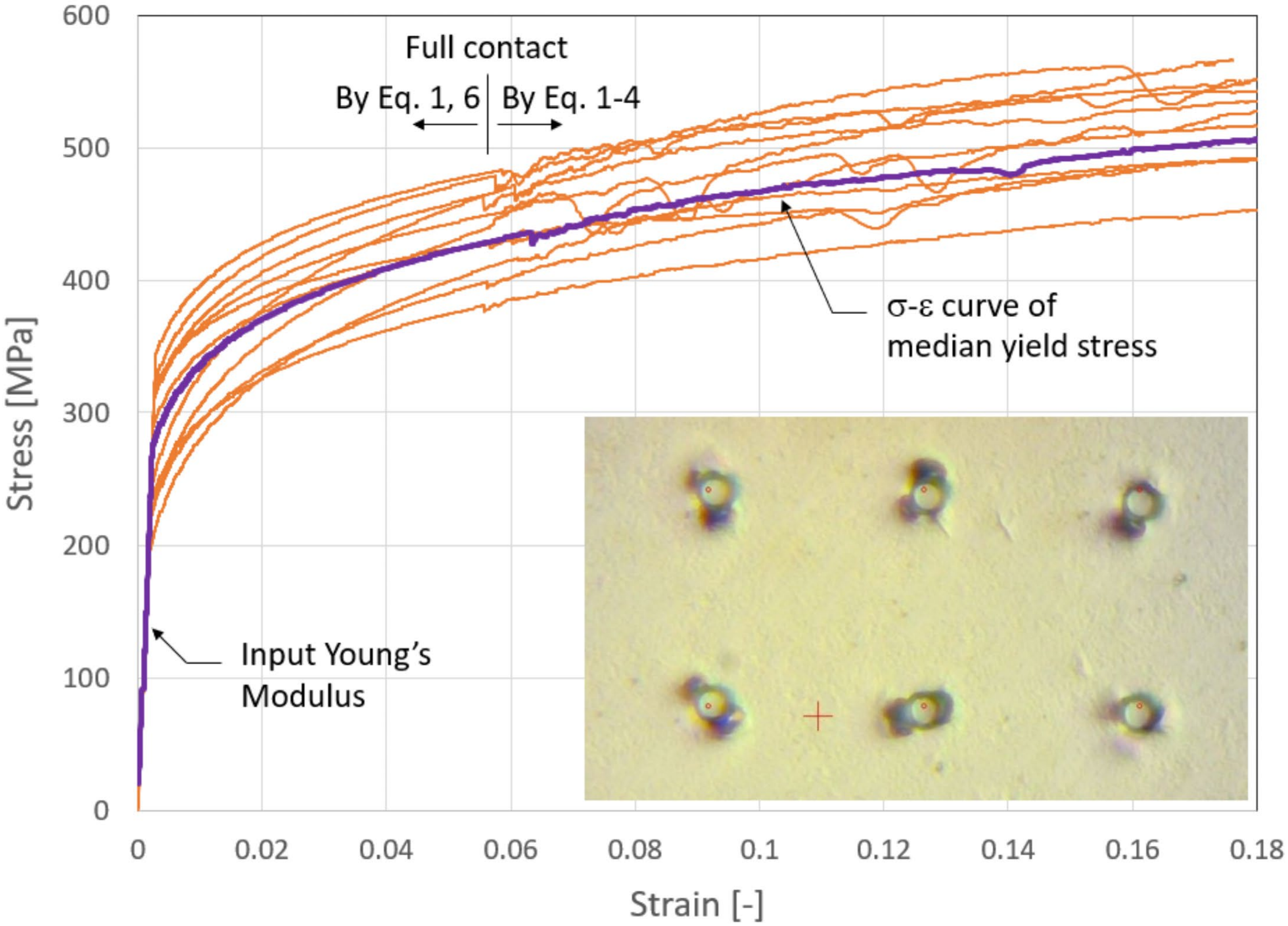

Figure 1. Stress-strain curves of pure titanium measured by 12 individual indentations with 10μm flat-ended punch. Inset: Six residual impressions (75μm spacing)

Points on the stress-curve prior to the achievement of full contact are calculated according to Equation 1 (for strain) and Equation 6 (for stress). The yield point is reported as the point of intersection between Equation 6 and the linear part of the stress-strain curve, which is set by the input Young's modulus.

Results and Discussion

The stress-strain curves for the twelve indentations are shown in Figure 1. Across all 12 tests, the yield stress was 270 ± 50MPa, which agrees well with the value of 240MPa measured by tensile test and reported by Kyocera[2], and is well within the range of values reported by others[3]. The scatter from test to test is higher than might be expected because each test is small enough to be confined to a single, randomly oriented grain.

Many others have endeavored to use a spherical indenter to measure small-scale stress-strain curves, but with limited success[7]. Spherical indentation includes several practical difficulties because the uncertainty in contact area is generally greater for spheres than for other indenter shapes. Furthermore, as the contact area grows, the volume of material being tested also grows, gradually and continuously incorporating virgin material into the test. Thus, both the material and strain are changing concurrently. With a flat punch, the contact area is well known and fixed as the area of the punch face. Because the contact area is fixed, the volume of tested material is roughly constant throughout the test.

Conclusion

The stress-strain relation of pure titanium was measured by nanoindentation using a flat-ended punch. The yield stress was 270 ± 50MPa. The flat-punch geometry is superior to the more commonly used sphere, because both the contact area and the volume of tested material remain constant throughout the test.

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