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Original Research Article

Elastic modulus of nanocrystalline titanium evaluated by cyclic tensile method

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The present study is devoted to examinations of the elastic modulus with the use of the uniaxial tensile test. The commertial purity titanium Grade 2 in the two states i.e. microcrystalline (mc-Ti) and nanocrystalline (nc-Ti) were examined. Bulk nc-Ti was fabricated by hydrostatic extrusion (HE) which is one of the severe plastic deformation methods (SPD). The elastic modulus of mc-Ti and nc-Ti were compared with the aim to analyze the influence of the nanostructure of titanium on its elastic modulus. The mc-Ti and nc-Ti samples were subjected to uniaxial tensile tests at various strain rates and various values of stress.

Generally, higher elastic modulus values were obtained in microcrystalline titanium. The elastic modulus of mc-Ti was evaluated at 107 GPa on average, whereas the elastic modulus of nc-Ti was 94 GPa on average. The nanocrystalline titanium had a lower elastic modulus than its microcrystalline counterpart by 13% on average, which can be attributed to the presence of significant volume of amorphous regions in the structure. Moreover, in this case a lower standard deviation of all the results was obtained. In most cases, with higher applied stress (load) the value of the modulus was lower, whereas at higher strain rates its value was higher.

1. Introduction

In the last years nanomaterials have been the subject of many investigations and publications [1]. One of the features of these materials, which is best examined and described, is their mechanical behavior [2]. It is commonly known that compared with their microcrystalline counterparts, nanomaterials are characterized by higher values of the yield stress, tensile strength, and hardness, whereas their plasticity and fracture toughness are lower [3]. Another basic parameter which describes the mechanical behavior of materials is their modulus of elasticity – *E*. In the case of nanomaterials there are however some problems with determining the elastic modulus. The possible relation between the value of this modulus and the structure of nanomaterials is difficult to describe. This is so since these materials are fabricated by various methods, various types of samples are prepared for their examinations, and various methods are employed for measuring their properties. That the results obtained thus far are not unequivocal can result from the fact that there are a variety of factors which affect the value of the elastic modulus in a given nanomaterial.

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In general, the value of the elastic modulus is defined by the three basic parameters: (1) forces of interactions between the atoms, which are associated with their equilibrium positions i.e. the equilibrium between the attractive and repulsing forces, (2) the bonding forces between the atoms, and (3) lattice constant of the elementary cell. Hence, in practice, the elastic modulus depends on the following factors:

(a) Chemical composition of the material, i.e. the purity and alloying elements. It has been found that, in titanium, the presence of oxygen, nitrogen, carbon, and aluminum results in an increase of the elastic modulus *E*, whereas vanadium and molybdenum decrease its value. It has been demonstrated that an increase of the Sn content in the Ti8VSn alloy results in a decrease of the elastic modulus, which is attributed to the lattice expansion of the material. [4]. Moreover, the elastic modulus appeared to depend on the kind, volume fraction, distribution, and morphology of the individual constituent phases [5], just as in the case of developed new type alloys intended for medical applications [6].
(b) Crystallographic orientation of a single grain, and, in anisotropic materials, the direction of action of the loading force. This is the case of pure titanium because of the difference in the constant of elementary cells of the lattice.
(c) Structural defects present in the material, such as the share of the grain boundaries in the material volume related to the grain size [7], the porosity, micro-cracks [8] and dislocation density. For example, Fougere et al. [9] suggested that the effect of porosity in decreasing the Young's modulus is the dominant structural feature. There are also studies which propose reduction of the Young's modulus by introducing pores [10]. The effects associated with structural defects are especially important in nanomaterials which contain great numbers of defects of various types. It should be noted that the occurrence and number of defects and, thus, the value of the elastic modulus, depend very often on the method employed for producing a given nanomaterial.
(d) Temperature of the material (i.e. the elastic modulus decreases with increasing temperature). The decrease of the elastic modulus during heating is due to an increase of the equilibrium interatomic distance. This is an effect of thermal expansion of material and weakening of the forces of interatomic bonds.

A method commonly known and long used for measuring the Young's modulus is the uniaxial tensile of the sample. The advantage of this method lies in that the tension is uniformly applied (along the measurement length) to the entire volume of the sample within its elasticity range. This method gives a macroscopic view on the properties of a relatively large volume of the material. In the present study, the volume of nanocrystalline titanium produced by hydrostatic extrusion was relatively large (i.e. bulk). The uniaxial tensile method, described above, permitted the present authors to determine the elastic modulus in the macroscopic scale, and at the same time to examine a relatively large volume of the nc-Ti.

The recent literature reports indicate that, at the present, the method most often used for measuring the Young's modulus is nanoindentation [6,7,11–13] whose main feature is that it examines samples with a relatively small volume. The nanoindentation method is also often used is surface engineering. As an example it can be mentioned the examinations of the Young's modulus of pure titanium after a modification of its surface described in ref. [11]. Huang et al. [12] used this method for measuring the Young's modulus of the nanoctructured surface layers produced by the surface mechanical attrition treatment (SMAT). The nanoindentation method has however a drawback in that the results can be false because of a possible adverse influence of the substrate on which the nanostructured layer is deposited, the anisotropy of the material, or local defects. Moreover, the result obtained by this method can also depend on the surface preparation procedure employed such as e.g. the mechanical treatment which generates stress and, in consequence, the strengthening of near-surface zone.

Other methods employed for measuring the elastic modulus include e.g. the free resonance vibration method [4], ultrasonic techniques [6], or electromagnetic acoustic resonance method, the latter being used for e.g. examining porous pure Ti and the Ti6Al4V alloy [10]. Cao et al. [14] determined the Young's modulus of nanocrystalline titanium produced by powder milling and consolidation by measuring the velocity of ultrasonic wave propagation.

It seems that the use of the uniaxial tensile method for measuring the elastic modulus of bulk nanocrystalline titanium has never been described in the literature. Therefore, the results obtained in the present study and their analysis will provide an innovatory contribution to the knowledge about nanometals.

Summing up there is the need to determine the Young's modulus of nanometals including the nanocrystalline titanium and the uniaxial method is proposed. The aim of this study was to examine the elastic properties of mc-Ti and nc-Ti and to determine the possible influence of the structure on the Young's modulus. The next goal was to explain what phenomenon causes possible differences. The study was also concerned with the effect of the strain rate and values of stress (i.e. load) applied during the tensile tests on the value of elastic modulus.

2. Materials and processing

The material investigated was commercial pure titanium Grade 2 (Table 1) in the two states: (1) as a microcrystalline titanium (mc-Ti), and (2) as a nanocrystalline titanium (nc-Ti). The nc-Ti was produced by plastic forming (i.e. hydrostatic extrusion – HE) of the mc-Ti (i.e. initial state). The mc-Ti material was delivered in the form of a rod with Ø33 mm. To produce nc-Ti, the mc-Ti rod was subjected to multi-pass HE process. The cold extrusion process was conducted at room temperature. The fabrication of nc-Ti was realized in 12 passes of extrusion during which the rod diameter was gradually reduced (i.e. Ø33 mm → Ø25 mm → Ø20 mm → Ø16 mm → Ø14 mm → Ø12 mm → Ø10 mm → Ø9 mm → Ø8 mm → Ø7 mm → Ø6 mm → Ø5.5 mm → Ø5 mm). In effect, nc-Ti was in the form of a rod with the Ø5 mm diameter (Fig. 1).

During each pass plastic deformation is induced in the material. The sum of deformation induced by all the extrusion