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PAPER • **OPEN ACCESS**

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To cite this article: A Schiavi *et al* 2018 *J. Phys.: Conf. Ser.* **1065** 062010

Comparison between tensile properties and indentation properties measured with various shapes indenters of Copper-Chromium-Zirconium alloy at macroscale level

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Abstract. In this paper the experimental results of tensile properties and indentation properties, as a function of pyramidal and spherical indenters, of Copper-Chromium-Zirconium alloy, in the macro-scale range at room temperature, are presented and compared. Measurements are performed on three Cu-Cr-Zr samples in order to evaluate different heat treatments: two samples are aged for 2 hours in a vacuum furnace at 480 °C, 550 °C and one sample is kept as received. The experimental procedures for the measurement of indentation modulus, by using the primary hardness standard machine at INRiM, and tensile modulus, by means of engineering tensile tests at CIRA, are described.

1. Introduction

Elastic properties of materials, in mechanical engineering and material science, can be evaluated by means of several different experimental techniques, based on static, quasi-static and dynamic methods. Observed elastic response may vary as a function of different measurement procedures and other boundary conditions, as a consequence some differences in elastic moduli can be easily achieved. Moreover the internal (microstructural) length-scale of the material system is also known to influence the measured mechanical properties. In order to quantify the differences in the elastic behaviors, a comparison at macroscale level between tensile properties and indentation properties, measured with indenters of different shapes, are presented. As it is well known, tensile modulus is the elastic response of a sample subjected to the action of a distributed load on a surface area, on the other hand the indentation modulus is properly the elastic response of the sample subjected to the action of a concentrated load in a single point. Occurring deformations in indentation tests are not linear, depending on the shape of indenter and on the indentation depth. This phenomenon, observed at micro- and nanoscale [1], is known as indentation size effect. In this work material under investigation is a Copper-Chromium-Zirconium alloy (chemical composition: 1%Cr, 0.06%Zr, rest Cu), with different thermal aging. Cu-Cr-Zr alloy is the primary candidate for structural, high heat flux components in future fusion reactors. This alloy is precipitation-hardened and the dominant length scale responsible for strengthening of the material is average spacing between Cr precipitates.
Tensile modulus is determined on the basis of stress-strain measurements, according to Standard methodologies (e.g., ASTM E8, ISO 10275, ISO 6892-1). Occurring deformations of the sample

subjected to a tensile stress, are measured by means of a newly developed optical technique (3D Digital Image Correlation) allowing to obtain a global and accurate high definition mapping of displacements and deformations, instead of local information.
Indentation modulus, as well as hardness, is determined from accurate measurements of indentation load, displacement, contact stiffness and hardness impression imaging. Measurements are performed with both pyramidal and spherical indenters, on the basis of ISO 14577-1 Standard and improved methodologies from literature [2-6].

2. Elastic properties

Elastic properties, in terms of tensile modulus E and indentation modulus E_{IT} of Copper-Chromium-Zirconium alloy samples, are evaluated in the macro-scale range at room temperature of 23±2°C.

2.1. Tensile modulus E

Tensile modulus E is calculated by dividing the measured incremental tensile stress $\Delta\sigma$ by the engineering extensional strain $\Delta\varepsilon$ in the linear (elastic) region of the occurring stress-strain curve, applying the classical Hooke's law at a constant strain rate, $E=\Delta\sigma/\Delta\varepsilon$. Plotting stress as a function of strain, the value of elastic modulus is determined by means of a linear fit, below the yield point σ_y, as shown below in Figure 1.

2.2. Indentation modulus E_{IT}

The method for measuring indentation modulus by indentation technique was introduced by Oliver and Pharr in 1992 [7]. Indentation modulus E_{IT} depends on several parameters and boundary conditions and it is expressed as:

$$E_{IT} = \frac{1-\nu_s^2}{\frac{2\sqrt{A_p}}{S\sqrt{\pi}} - \frac{1-\nu_i^2}{E_i}} \tag{1}$$

where ν_s is the Poisson ratio of tested material, ν_i and E_i are the Poisson ratio and the Young's modulus of the indenter material, S is contact stiffness, i.e. the incremental ratio between unloading force and related displacement at maximum depth of indentation and A_p is the projected contact area, i.e. the value of the indenter area function at the contact depth.
The projected contact area A_p depends on the depth h_c of the contact of the indenter with the sample at F_{MAX} and on type of indenter: for Vickers diamond pyramidal indenter, with a vertex angle α, $A_p=(2h_c\cdot\tan\alpha/2)^2$ and for Brinell tungsten carbide spherical indenter, with a radius R, $A_p=\pi h_c\cdot(2R\text{-}h_c)$.
In both cases, the depth h_c of the contact of the indenter with the sample at F_{MAX}, is determined as a function of frame compliance C_f, as follows:

$$h_c = h_{\text{MAX}} - \varepsilon \cdot \frac{F_{\text{MAX}}}{S} - C_f \cdot F_{\text{MAX}} \tag{2}$$

in which h_{MAX} is the maximum indentation depth, F_{MAX} is the maximum of applied force, ε is a value depending on the indenter geometry and the extent of plastic yield in the contact (for both Vickers and Brinell ε=0.75), S is the contact stiffness and C_f is determined, for each single measurement as a function of maximum experimental indentation depth h_{MAX} and the indentation depth h_v measured from the actual hardness impression on the sample [4], i.e., $C_f= (h_{\text{MAX}}\text{-} h_v)/F_{\text{MAX}}$.
In particular for Vickers indenter h_v=0.5l·cot(0.5α), in which l is the side length measured from the actual hardness impression, and for Brinell indenter $h_v=R\text{-}(R^2\text{-}r^2)^{1/2}$, in which r is the measured radius of the resulting hardness impression on the sample.

In Figure 1 the experimental data needed for the tensile and indentation modulus determination are depicted: a typical experimental stress-strain curve, with the linear (elastic) region below the yield strain value σ_y, and an experimental loading-unloading curve of indentation test, as a function of applied force F and occurring displacement h. A typical Vickers hardness impression, with measured diagonal d and side length l, is also shown.

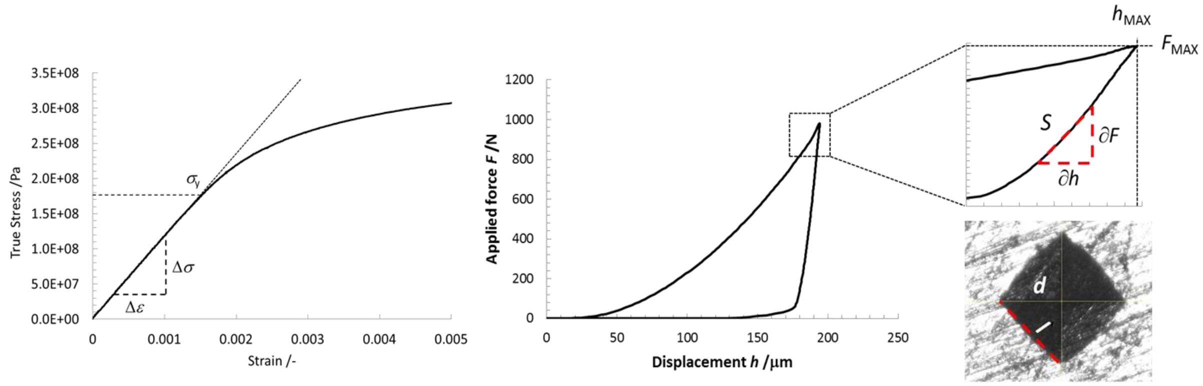

Figure 1. Typical experimental stress-strain curve and loading-unloading curve of indentation test.

3. Experimental results and comments

Tensile modulus of Cu-Cr-Zr alloy is determined from uniaxial tensile tests by means of an Instron 4505 universal testing machine with a 50kN load cell installed. The displacement rate is set to 0.1 mm/min. Strain is measured using a 3D Digital Image Correlation system. Geometrical dimensions and shape of the samples are in accordance with proportionality stated in international Standards.

Indentation modulus (and hardness) is determined from both Vickers and Brinell procedures, by applying the typical loads (corresponding to the maximum of applied force, F_{MAX}) recommended for hardness tests, i.e., 3 kg, 30 kg and 100 kg for Vickers and 31.2 kg, 62.5 kg and 187.5 kg for Brinell. Brinell tests are performed by using two spherical indenter (diameter D_1=2.5 mm and D_2=1.0 mm).

Occurring maximum indentation depth h_{MAX} is measured by a laser interferometric system. Moreover the average diagonal d_{av} and the calculated square side l for Vickers impressions, as well as the average diameter d and the calculated radius r for Brinell impressions, used for the evaluation of hardness impression depth h_v, are measured from optical microscopy. Contact stiffness $S=\partial F/\partial h$ is evaluated on the basis of Doerner-Nix linear model [8].

In Table 1 the experimental results of tensile test, in terms of tensile modulus E and yield stain σ_y and hardness test, in terms of Vickers hardness HV and Brinell hardness HBW, are shown.

Table 1. Elastic properties and hardness values of Cu-Cr-Zr samples treated with different aging.

	E (GPa)	σ_y (MPa)	HBW D=1.0mm	HBW D=2.5mm	HV 100	HV 30	HV 3
Id0 (no aged)	95.1	44.7	75.2	62.9	57.8	62.7	90.0
Id3 (aged 480 °C)	115.4	175.3	134.0	129.1	134.7	139.2	147.7
Id5 (aged 550 °C)	107.7	101.8	118.5	110.8	111.8	115.6	134.8

As it is possible to notice from HV values, an increase of the hardness by decreasing the applied load (and as a consequence the indentation depth), can be observed, such as a kind of indentation size effect [8-12], also at macroscale level. On the other hand, for HBW values, similar effect is not achieved, as a function of load, but as a function of indenter radius, as observed from experimental results at microand nanoscale [1]. A similar behavior can be observed from values of the indentation modulus E_{IT}, as shown in graphs of Figure 2. It is important to underline that the effects due to the frame compliance C_f (sample compliance C_s is negligible) are taken into account, in the experimental results, by using relation (2). On average measured frame compliance C_f ranges between 100 nm/N and 10 nm/N, depending on applied loads.

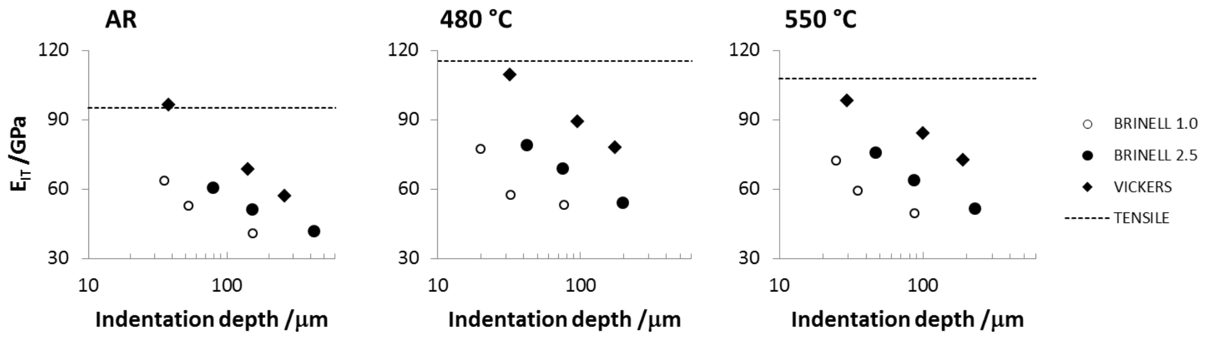

Figure 2. Comparison between indentation modulus E_{IT} and tensile modulus E (dotted line) of CU-Cr-Zr alloy samples expressed as a function of measured indentation depth h_{MAX}.

4. Conclusion

A comparison between tensile and indentation properties at macroscale level, of 3 samples of Cu-Cr-Zr alloy, previously aged at different temperatures, is presented. The Cu-Cr-Zr alloy is the primary candidate as a component in future fusion reactors. Tensile modulus is determined on the basis of stress-strain measurements. Indentation modulus (and hardness) is determined from both Vickers and Brinell tests, by applying standard methods and an improved procedure, allowing to avoid effects due to the frame compliance during the test. Experimental results show systematically that the elastic response is indentation depth dependent, as well as load dependent. This behavior is generally observed at micro- and nanoscale level and it is defined in terms of indentation size effect.

5. Acknowledgements

The research leading to these results is conducted in the frame of EMPIR 14IND03 “Strength-ABLE”. The EMPIR is jointly funded by the EMPIR participating countries within EURAMET and the European Union.

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