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Effect of neighboring phase properties on measured indentation data

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Abstract. The paper focuses on the variation of mechanical properties near the interface of two materials. The aim is to determine the properties of the individual phases and the size of the region influenced by the adjacent material. Nanoindentation measurements were performed in the vicinity of a sharp interface between tungsten and steel, which was a plane located parallel to the loading force direction. The probability of indentation in the affected area as a function of depth of penetration and distance from the interface was fitted by a proper statistical distribution function. The intrinsic properties of the individual phases can subsequently be extracted from the experimental indentation data.

Introduction

Tungsten is a prime candidate material for a first wall of tokamaks, which may serve as future fusion reactors. For joining of tungsten to the support structure and the cooling system, composites or graded layers may be used to reduce the stress concentration compared to a sharp interface. Mechanical properties of such composites, as well as of the individual phases, are of interest. Nanoindentation is a popular method that can be used for this purpose, one of its features being the very local character of the measurement. To get a statistically significant amount of data, a matrix of indents is often used, which can be pre-programmed and thus does not need the operator's control for every indent. In the case of a composite, such indents will have random positions with respect to the individual phases and their interfaces. Near the interface, the indentation response is affected by both phases present, because the stressed zone under the indenter spans across the interface The goal of this study is to determine the Young's moduli of the pure materials from matrix indentation data and the progression of the modulus change near their interface.

Method

Experimental sample consisting of tungsten and steel bi-layer was prepared using spark plasma sintering (SPS) from pure tungsten and steel powders. The specimen had a sharp macroscopic interface, both phases have Poisson ration ν of 0.3. A matrix of indents was applied on a section perpendicular to the interface in its vicinity. Continuous multi cycle (CMC) loading with 5 increasing load levels ranging from 10 to 50 mN was used to obtain Young's modulus at different distances x from the interface. Young's modulus values were calculated using the Oliver-Pharr method [1]. All distances were normalized by penetration depth h_c, assuming in the first approximation that the indents are self-similar [2]. The aim of the consequent statistical analysis was to find the influence of the size of the area D covered by indents on its fraction p_3 in which indentation moduli are affected by the material interface. .

In the first step, the dependence of Young's modulus, E, on the normalized indenter-interface distance x/h_c was approximated by cumulative distribution function (CDF) of symmetric ($\alpha = \beta$) inverse beta distribution [3, 4, 5]. The purpose was to determine Young's modulus of pure tungsten and pure steel. The boundary between pure phases and the interfacial region was considered as 2% deviation from Young's modulus of the pure phase.

In the second step, the aim was to obtain the parameters of beta distribution with probability distribution function (PDF) given by Eq. 1 from a histogram of E.

$$pdf(E) = \frac{\left(\frac{E - E_1}{E_2 - E_1}\right)^{\alpha-1} \left(1 - \frac{E - E_1}{E_2 - E_1}\right)^{\beta-1}}{(E_2 - E_1)\mathrm{B}(\alpha, \beta)} \quad (1)$$

This represents the simulation of indentations on a composite with random positions with respect to the phase boundary. The task is more difficult because there is no information about the indenter-interface distance in the histogram. For the histogram of measured Young's modulus of a tungsten-steel component, a class size of 5 GPa was selected. For fitting of the histogram, a MATLAB program was established. The Gauss distributions of the pure phases' moduli were searched at first. The remaining data, representing the region influenced by both phases, were approximated by a probability distribution function (PDF) given by conditional probability of mean values of pure phases' Young's moduli with Gauss distributions integrated in beta distribution.

Results

Sets of a data measured up to the maximal indenter-interface distance D were normalized by the relevant penetration depth h_c. The dependence of Young's modulus on a normalized indenter-interface distance was approximated by CDF of inverse beta distribution (see Fig. 1, left). This way, Young's moduli of pure tungsten and pure steel were determined, taking 2% deviation from pure phase values for the boundary of the interfacial region (influenced by the other phase).

The parameter α (=β) was found to be practically independent on the set of indents delimited by D (see Tab. 1). The results are shown in Fig. 1. The fitting results on the left are checked by a histogram on the right. Gauss distributions were calculated from areas which were assumed as pure phases. Cumulative distribution function of beta distribution is in the left part of Fig. 1, its probability distribution function is on the right. We can note a good agreement with the experimentally determined histogram of E. Tab. 1 summarizes the relevant parameters of the approximation.

The main task is a reverse process – to obtain the properties of the pure materials and the variation in the interfacial region from the histogram. The results of the identification on simulated microstructure are shown in Fig. 2 and in Tab. 2. The accuracy of the identified Young's moduli is within 5 GPa, which is the size of the histogram bin. The actual size of the area affected by indentation on the interface p_3 remains practically the same for the all simulated considered areas, which proves the identification methodology.

Summary

The mechanical properties of pure phases and the interfacial region of a tungsten-steel composite were investigated in this study, using nanoindentation and modeling with selected statistical functions. The goal was to determine the mean values and variances of pure phases' Young's moduli from its simulated histogram. The values in pure phases were approximated by Gauss distributions and the values in the interfacial region were approximated by beta distribution. The parameters (Fig. 2, Tab. 2) obtained this way show a good agreement with the parameters obtained from the Young's modulus dependence on the normalized indenter-interface distance (Fig. 1, Tab. 1).

Tab. 1. Parameters corresponding to Fig. 1. *D* (x_{max}/h_c) is the considered area around the interface, $p_{3,norm}$ is the normalized size of the interfacial region, $p_{1,norm} + p_{2,norm} + p_{3,norm} = 1$, E_1 and E_2 are mean values of pure phases (tungsten, steel), σ_1 and σ_2 are the variances of pure phases, $\alpha=\beta$ are parameters of the beta distribution.

D	$p_{3,norm}$	E_1 [GPa]	E_2 [GPa]	σ_1 [GPa]	σ_2 [GPa]	$\alpha = \beta$
60	0.18491	242.26	386.46	12.139	26.602	0.54963
80	0.14474	241.42	379.84	11.247	28.586	0.54149
100	0.11792	241.21	378.7	11.865	28.768	0.53841
120	0.10724	241.02	377.93	11.456	28.795	0.54538

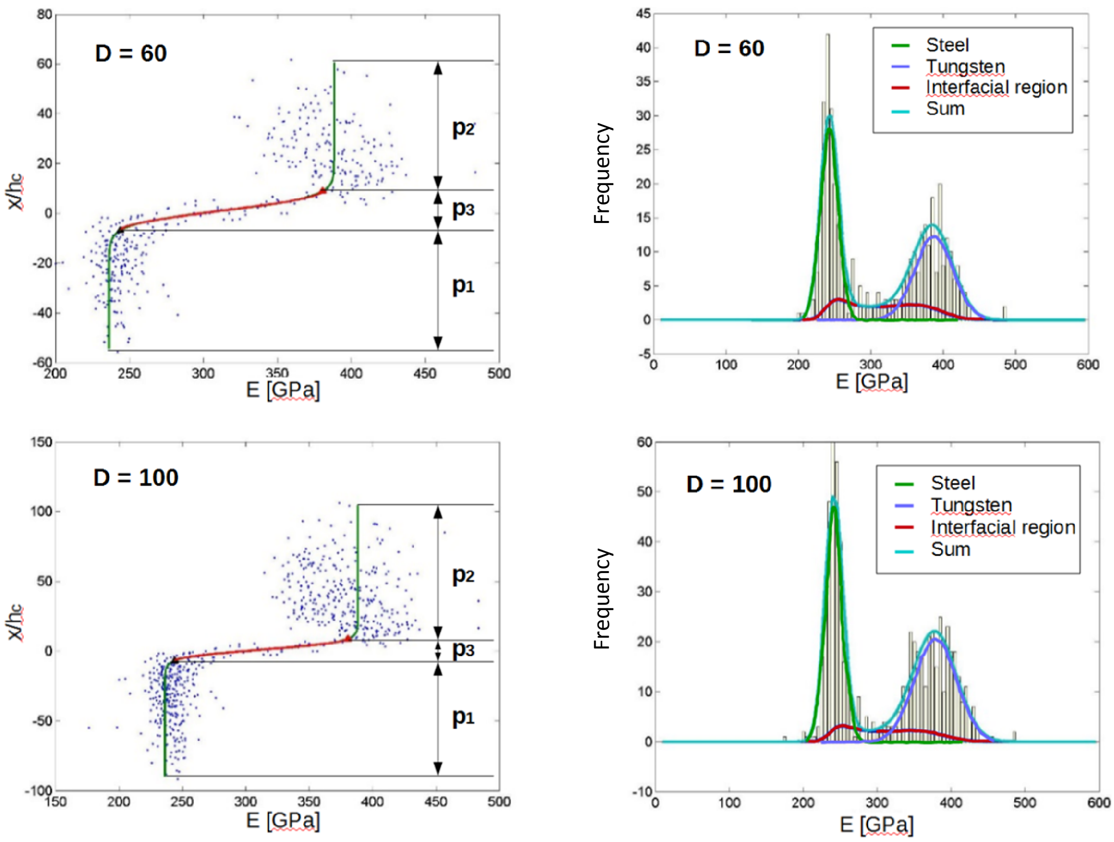

Fig. 1. Left: Approximation of Young's modulus dependence on a normalized distance. The area marked p_2 corresponds to pure tungsten, p_1 represents pure steel; the variances of the respective Young's moduli are calculated from the data in these areas. Parameter p_3 corresponds to the area fraction of interfacial region. The data are approximated by beta distribution to find the mean values of Young's moduli and the variation in the interfacial region. Right: A check of the approximation - the sum of probability distribution functions of two Gauss distributions and beta distribution compared with experimental *E* histogram.

Tab. 2. Parameters corresponding to Fig. 2.

D	$p_{3,norm}$	E_1 [GPa]	E_2 [GPa]	σ_1 [GPa]	σ_2 [GPa]	$\alpha = \beta$
60	0.20544	240	385	9.2126	24.798	0.59178
80	0.14908	240	380	10	30	0.5
100	0.11786	240	380	10.326	30	0.44607
120	0.10544	240	380	10.422	30	0.36677

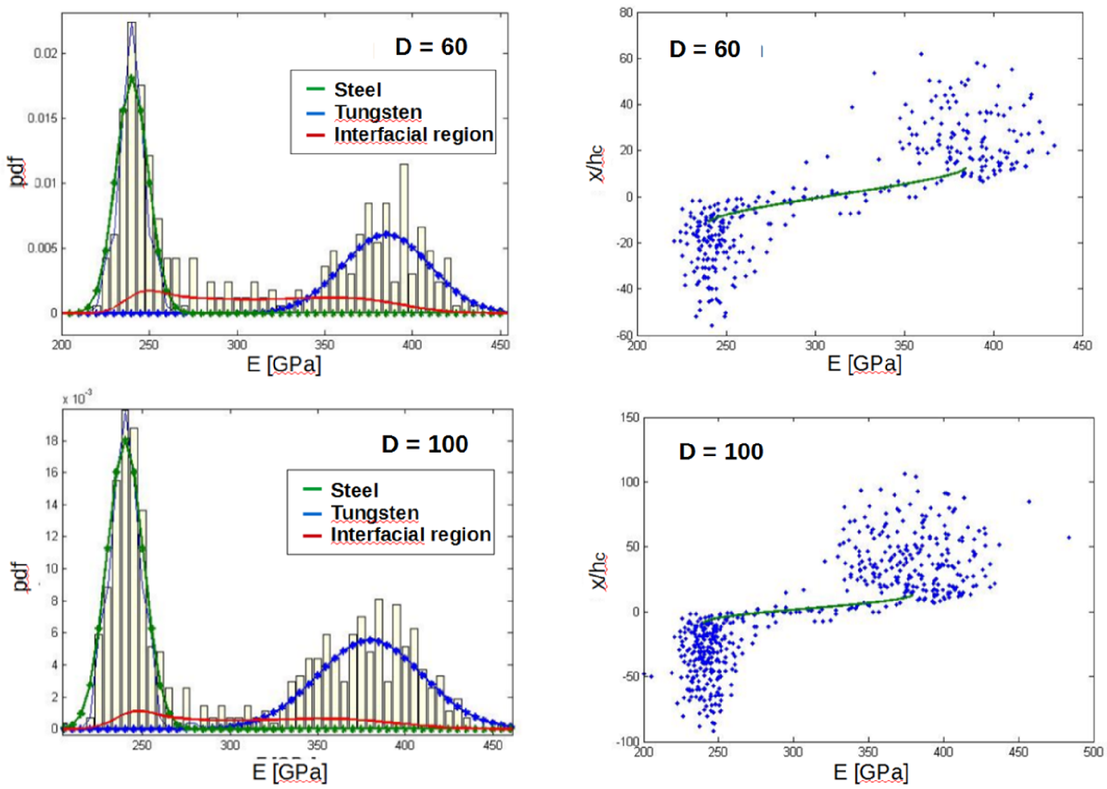

Fig. 2. Left: a histogram of measured parameters, approximated by a mixture of two Gauss distributions and beta distribution without the knowledge of x and p_3. Right: a check of the results; CDF of the obtained inverse beta distribution plotted with the experimental data, spanning between the mean values of Young's moduli of steel and tungsten.

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