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GRID NANOINDENTATION ON MULTIPHASE MATERIALS FOR MAPPING THE MECHANICAL PROPERTIES OF COMPLEX MICROSTRUCTURES

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Abstract – Instrumented indentation has now become established for the single point characterization of hardness and elastic modulus of both bulk and coated materials [1-3]. This makes it a good technique for measuring mechanical properties of homogeneous materials. However, many composite materials are composed of several phases whose properties are difficult or even impossible to examine in bulk form ex situ (e.g., carbides in a ferrous matrix, martensite and austenite in steels, etc.). The requirement for in situ analysis and characterization of such structured materials with different phases obviates conventional mechanical testing of large specimens' representative of these material components. This paper will focus on new developments in the way that nanoindentation can be used as a two-dimensional mapping tool for examining the properties of constituent phases independently of each other. This approach relies on large arrays of nanoindentations (known as grid indentation) and statistical analysis of the resulting data. Two examples of application of the grid indentation method will be presented: indentation on naval brass and indentation on AISI 301 stainless steel.

Keywords (up to three): grid indentation, multiphase materials, statistics

1. INTRODUCTION

Instrumented indentation or shortly nanoindentation is very powerful technique for characterization of materials in small volumes. Despite its undoubted contribution to the measurements of mechanical properties care must be taken during experiments to properly understand the obtained results while testing certain types of materials.

Among such materials belong especially non-homogeneous (multiphase) materials whose indentation testing is complicated for several reasons:

(a) When indenting single phases or grains, it is impossible to know the depth of the phase below the surface, so even a relatively shallow indent may be sensing the other phase's properties.

(b) Very often a compromise has to be made between having sufficient indentation depth to overcome surface roughness (even despite polishing procedure), but shallow enough to measure the phase-only properties.

(c) Individual phases are not always visible by microscopy, or no suitable etchant is available.

However, several recent studies [4-7] showed a new method, which overcomes the above mentioned obstacles by using a large number of indentations and their statistical treatment. This method, called grid indentation, is based on large matrices of indentations (~several hundreds) and subsequent statistical analysis of the indentation results. Figure 1 shows the principle of the method; its theoretical background is in detail explained in [5, 6].

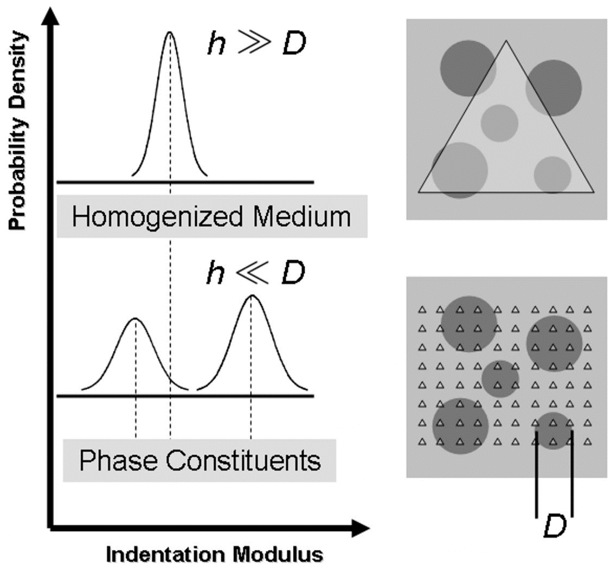

Fig. 1. Schematic of the principle of the grid indentation technique for heterogeneous materials. Small indentations allow the determination of single phase properties while larger indentations yield the properties of the homogenized medium. *D* is the characteristic dimension of the phase and *h* is the indentation depth (adapted from Constantinides et al., see Ref. 5).

Basically, a large number of indentations is performed and automatically analyzed to obtain hardness, elastic modulus and eventually other mechanical properties. Such property (for example hardness) is then plotted in form of a frequency plot (normalized histogram), which is then fitted by a number of probability density functions (PDF) [7, 8].

For most applications, Gaussian distribution can sufficiently well describe the distribution of the experimental results. In a special case of two phase material, the bimodal Gaussian distribution of hardness is given by

$$f = \frac{1-p}{\sqrt{2\pi}\sigma_1}\exp\left[-\frac{(H_{IT}-\mu_1)^2}{2\sigma_1^2}\right] + \frac{p}{\sqrt{2\pi}\sigma_2}\exp\left[-\frac{(H_{IT}-\mu_2)^2}{2\sigma_2^2}\right] \quad (1)$$

Where H_{IT} is the hardness, p is the mixing parameter and μ_1, σ_1, μ_2, σ_2 are the parameters of the distribution. These parameters then represent the corresponding property of the given phase and its standard deviation, i.e. μ_1 is the average hardness of phase 1 and σ_1 is the standard deviation of hardness of the same phase.

2. EXPERIMENTAL

Application of this indentation technique is demonstrated on two materials:

(a) Naval brass (CDA 464) with composition of Cu-39.2 Zn-0.8 Sn, which contains two primary phases, α and β, whose mechanical properties are known to be very similar and indistinguishable by standard microindentation techniques. The characteristic dimensions of each phase (grain size) were approximately 24 μm. The grid indentation was performed in form of a 20 × 20 indents matrix, where individual indentations were spaced by 5 μm. Maximum load was 2 mN with a 15 s hold to eliminate creep. Maximum indentation depth was between 150 nm and 200 nm. The goal of the grid indentation was to determine hardness and elastic modulus of both α and β phases.

(b) Bright annealed AISI 301 austenitic steel with (in wt.%) 0.05 C, 17 Cr, 7 Ni, 0.5 Si and 0.1 Mo chemical composition. This steel is known to undergo deformation-induced phase transformation of face-centered cubic (fcc) γ austenite to body centered cubic (bcc) α'-martensite. For more details on this phase transformation see [9-11]. Three samples from this steel with different strain levels were tested: 0 % (undeformed state), 10 % deformation and 20 % deformation. The undeformed sample contained solely austenite, 10 % deformed sample contained predominantly austenite with some fraction of martensite and the 20 % deformed sample contained predominantly martensite with remaining fraction of austenite. The grid indentations were performed on two randomly selected areas by 20 × 20 matrix with 5 μm spacing. The maximum load was set to 1 mN which yielded maximum indentation depth of ~100 nm. To eliminate creep the maximum load was held constant for 5 s. The grid indentation was used on this material to determine the mechanical properties of austenite and martensite, which would be extremely difficult to determine by single load indentations as the martensite is formed in form of laths with micro- and submicrometric diameter.

All indentation measurements were performed on a CPX-NHT Nanoindentation Tester (CSM Instruments, Peseux, Switzerland) with a Berkovich diamond indenter using instrumented indentation technique. The NHT uses passive top referencing concept which eliminates to a great extent the negative effects of thermal drift of the instrument and it is therefore perfectly suited for such long term measurements (a complete measurement of a matrix containing 400 indentations typically takes about 10 hours). The large volume of resultant data is then exported, plotted as histograms, and deconvoluted to extract property and volume fraction information. All indentation tests were done in compliance with the ISO 14577 standard, as was the subsequent calculation of indentation hardness and elastic modulus.

2. RESULTS

2.1. Naval brass CDA 464

The results of the grid indentation on the naval brass are shown in fig. 2 in form of hardness (fig. 2a) and elastic modulus (fig. 2b) histograms. These histograms were fitted by bimodal Gaussian distribution according to equation (1) and the parameters of the distribution were calculated according to procedure published in [5]. For comparison of the indentation response on the two phases two typical indentation load-displacement curves are shown in fig. 2c.

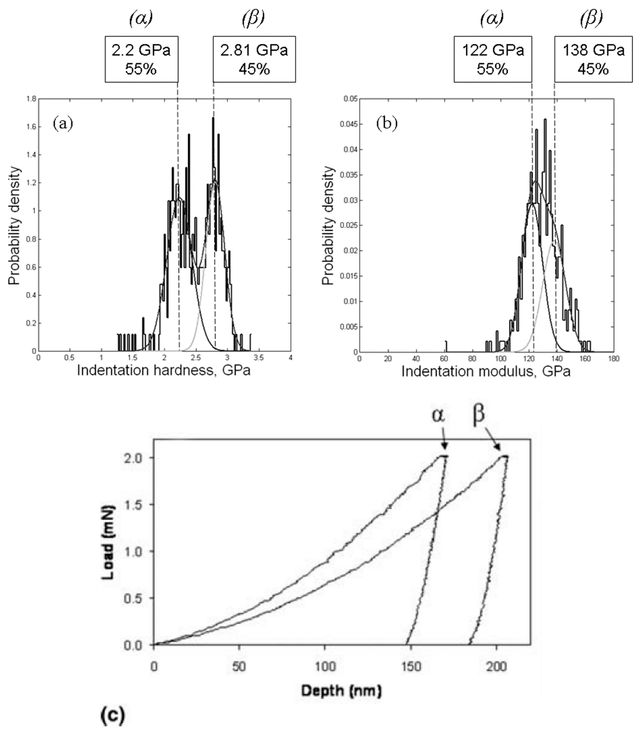

Fig. 2. Grid indentation histograms of hardness (a) and elastic modulus (b) on the naval brass with the corresponding bimodal fits and their parameters. Typical indentation load-displacement curves (c) show the difference in indentation response of both α and β phases. Numbers in percents indicate the volume fraction of each phase.

Two distinct peaks are visible in Fig 2a: one peak corresponding to α-phase with hardness of 2240 MPa and standard deviation of 200 MPa and another peak corresponding to β-phase with hardness of 2810 MPa and standard deviation of 150 MPa.

The difference in elastic moduli of the two phases is less pronounced and the peaks are closer (see fig. 2b): the first peak corresponding to α-phase with mean elastic modulus of 122.3 ± 7.5 GPa and the second peak corresponding to β-phase with mean elastic modulus of 138.0 ± 8.2 GPa.

2.2. AISI 301 austenitic steel

The results of grid indentation obtained on the AISI 301 steel with three deformation levels in form of hardness histogram are shown in fig. 3. One histogram is plotted for each deformation level. The parameters of the distribution were identified by maximum likelihood method using Generalized Reduced Gradient nonlinear optimization [12]. Although the peaks for austenite and martensite were overlapping, the fitting method was robust enough to distinguish two peaks in the case of the 10 % and 20 % deformed samples.

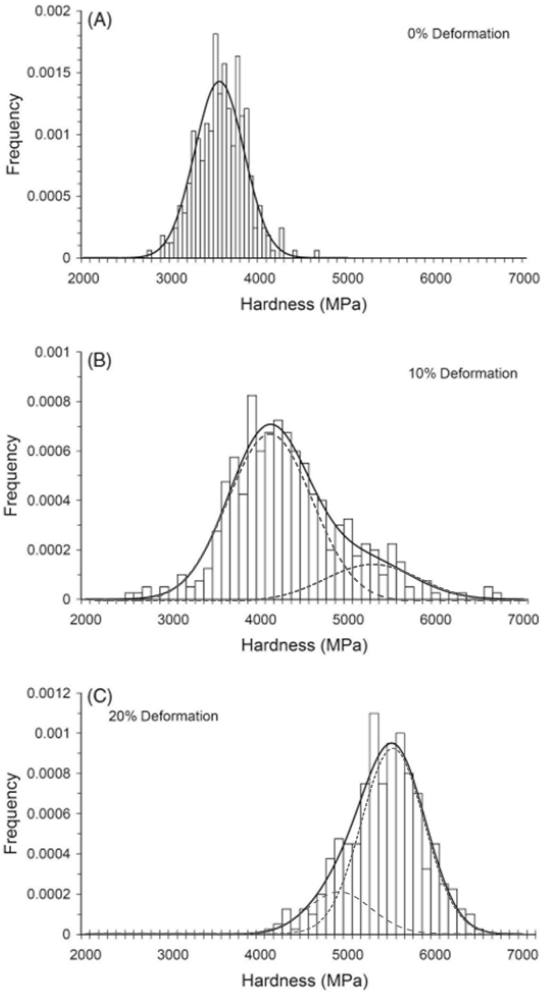

Fig. 3 Hardness histograms for grid indentation results on AISI 301 austenitic steel: (a) non deformed state, (b) 10 % deformation, (c) 20 % deformation.

The bimodal Gaussian fit of the 10 % deformed sample revealed hardness of austenite of 4043 ± 461 MPa and martensite hardness of 5155 ± 517 MPa with volume fraction of martensite of 21 %.

Similarly, bimodal Gaussian fit of the 20 % deformed sample revealed austenite hardness of 4867 ± 368 MPa and martensite hardness of 5487 ± 361 MPa with volume fraction of martensite of 81 %.

As for the sample in the non deformed state, the bimodal Gaussian fit showed existence of only one phase (austenite) as expected. Hardness of this unique phase was 3539 ± 269 MPa.

3. DISCUSSION

The two case studies presented in this paper were selected to demonstrate the possibilities of the recently developed grid indentation technique. All indentations in the grid indentation array were load controlled, so the maximum load was identical for all measurements in the given array. The resultant indentation depths were therefore directly linked to the mechanical properties of the individual phases and varied depending on whether the particular depth corresponded to a pure phase or an interaction between the two phases. In the particular case of two phase materials, there were three possibilities of the location of the indent: 1) indent in phase one, 2) indent in phase two and 3) indent on a boundary between the two phases. To ensure that the properties of only one phase could be measured, the maximum indentation load was carefully selected so that the maximum indentation depth and the corresponding volume involved in the material's response reflected the properties of a single phase. In general, the maximum indentation depth is set so that $h/D < 0.1$ where h is the indentation depth and D is characteristic dimension of the phase or grain. This rule was respected in all presented measurements. Higher indentation depth would result in an overall mechanical answer of the material and it would be impossible to measure properties of individual phases even in case when the indent is located on only one phase. On the other hand, the indentations were sufficiently deep so that the negative effect of surface roughness on the scatter of the data was greatly reduced.

The selection of the area to indent is very important as the grid indentation usually includes square areas of several hundreds of micrometers large or less. Therefore the area selected for testing must be representative of the whole material. The advantage of the statistical analysis of the grid indentation results to estimate the volume fraction of given phase also underlines the importance of proper selection of the indented region.

The surface of the sample must be prepared by polishing to the lowest roughness possible, however without the effect of work hardening or pulling out of purely bonded particles. If work hardening or residual stresses are expected to be formed during polishing, other methods of surface preparation such as electrolytic polishing shall be considered. This was the case of the AISI 301 steel which was electro-polished in 5 % perchloric acid solution in ethanol at 40 V to avoid formation of a surface layer affected by mechanical grinding and polishing, which could itself introduce the martensitic transformation.

The application of the grid indentation technique on the two selected materials showed that this method can be used not only for materials where distinct peaks in the histograms are found but also for materials where the peaks are closely overlapping. In the first example (naval brass) two distinct hardness peaks in the hardness histogram were found and the fit results could be approximately verified by superposing both distribution functions over the obtained histogram. However, in the case of AISI 301 steel, only one peak was visible on the hardness histogram and such empiric verification could not be done. Thanks to the robustness of the fitting algorithm, two distribution functions were found and when superposed on the hardness histogram, one could verify that the two distributions actually fit the original histogram.

A very simple indication of the presence of two distributions in the histogram on the 10 % and 20 % deformed samples was that the histogram was either right-tailed (10 % deformed sample) or left-tailed (20 % deformed sample). The right-tailed hardness histogram generally means that it is composed of a predominant distribution function with lower mean hardness and a lower volume fraction of distribution function with a higher mean hardness. Left-tailed hardness histogram generally means that it is composed of predominant distribution function with high mean hardness and a lower fraction of distribution function with a lower mean hardness. This was confirmed by the estimation of volume fractions and mean hardness of the martensite in the 10 % and 20 % deformed samples, where the volume fraction of martensite increased from 21 % in the 10 % deformed sample to 81 % in the 20 % deformed sample. Correspondingly, hardness of martensite was in both cases higher than hardness of the austenite.

As found by the Gaussian fits of the 10 % and 20 % deformed samples, hardness of austenite was increasing with the level of deformation. This is probably due to the fact that the austenite was constrained by the growing number of martensite laths, formed by the deformation induced transformation. Consequently, the grid indentation method can also be sensible to changes in structural arrangements of the material.

The Young’s modulus histogram of the AISI 301 steel was not for the statistical analysis as the histograms for all three samples were very symmetrical indicating very similar mean values of Young’s modulus of austenite and martensite. Indeed, Young’s modulus of austenite and martensite of similar materials are known to be very close to a common value of 200 GPa.

4. CONCLUSIONS

This paper showed the principle and application of a recently developed grid indentation technique for analysis of mechanical properties of multiphase materials. This technique is based on repeated indentation in form of a rectangular matrix with a total indentation count up to several hundreds. The results of each indentation are calculated automatically and the resulting data set is then analyzed statistically, which allows determination of the mean hardness and elastic modulus of each phase and their volume fraction.

The technique was demonstrated on two case studies. The first study was focused on a simple application of the technique and discussed general parameters and conditions of this indentation technique. The second case study showed application of this method on a more complex material with phase transformation and where so far other methods failed to determine mechanical properties of the two phases.

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