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# An application of universal hardness test to metal powder particles

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ABSTRACT

Powder metallurgy is a "net shape" components producing technology from metal powders by compaction with following sintering processes. For today actual trends of powder metallurgy are associated with modern powder grades, alloyed by elements with high affinity to oxygen (Cr, Mn, Si, etc.). Contamination of powder particles by oxides and/or other secondary phases have a negative effect on their compressibility and sinterability. The geometry properties of powders give integral information about powder quality. Evaluation of yield strength and/or rather the strain hardening exponent, characterizing the mechanical properties on the level of individual particles, really is not possible. One of available approaches could be measurement of the microhardness of particles. The contribution deals with the evaluation of the microhardness of powder particles and specification of the factors affecting measured values. Using standard Vickers microhardness HV0.01 measurements for two different powders the results obtained showed large scattering from the average. This gave no possibility to identify the influence of alloying and particle matrix purity on microhardness. Problem was solved utilizing instrumented indentation test using NanoIndenter XP. This is usable technique for estimation of microhardness of powder particle matrix and gives possibility to recognize differences between different size fractions of particles. Based on the obtained results it was concluded, that absolute results of indentation hardness and indentation modulus are strongly affected by mounting resin type. Utilizing DSI method and mounting resin of proper hardness enabled to evaluate the microhardness of powders with different alloying element content. Influence of particles purity/size on powder microhardness was established as well. Indentation hardness and indentation modulus for sintered materials are in good agreement with the data for corresponding bulk materials. Obtained results confirm that universal hardness test is valuable instrument for evaluating of sintered materials properties.



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## 1. Introduction

The aim of powder metallurgy processes is to produce a green compact from a metal powder with sufficiently high density and green strength. Further processing by sintering at suitable conditions (temperature, time, atmosphere) gives a sintered part with defined mechanical properties (German, 1994; Höganäs Handbook, 2004).

Metal powder represents a statistical set of particles with different geometrical (size distribution, shape, specific surface) and mechanical (hardness, yield strength, strain-hardening rate, etc.) properties of particle material. The change from powder to compact under pressing pressure can be interpreted by different overlaying mechanisms. For low pressures the densification of the powder occurs by the movement of particles – translation and rotation. With pressure increasing contact area between the particles increases and particles undergo extensive plastic deformation. This is associated by work-hardening resulting in diminishing of the densification rate. This stage of compaction is governed by the ability of the metal matrix of compact to plastic deformation development, it means by yield strength of particle materials. In the event of unsuitable particles microstructure, eventually wasted by oxides or other secondary phases, the lower compressibility and frequently also sinterability of powders occur (Parilak et al., 2004). The geometry properties of powders are tested by standard methods giving integral information about powder quality. Meanwhile evaluation of the yield strength and/or rather the strain hardening exponent, characterizing the mechanical properties on the level of individual particles, really is not possible. One of available approach would be particle microhardness measurements, as was described by Parilak et al. (1983).

This contribution deals with the evaluation of powder particles mechanical properties and specification of effects affecting measured results. This problem arouses up by Vickers microhardness HV0.01 measurements that were made in the dissimilar part of the particles and in the particles of various sizes. Powder particles were mounted in acrylic mounting resin and testing was performed using LECO LM700AT equipment, see Fig. 1. From Figs. 2 and 3, it is evident that powders exhibit different values in the same particle resulted in a large deviation of the average value. This is due to the fact that the properties of microstructure, particle size, etc., have much more pronounced influence on the results of microhardness measurements, than alloying. Large deviations of the results from the average values is associated mostly with three facts – method of the measurements, influence of mounting resin properties and "depth" of the particle under indenter (cutting effect). This influence can be removed if microhardness of the particles is estimated by depth sensing indentation (DSI) (Zubko et al., 2005).

## 2. Theoretical description

Hardness is defined as the resistance of a solid material against the penetration of another harder material into its surface. This definition is not completely fulfilled for the usual hardness test methods for metallic materials as Brinell, Vickers and Rockwell. This is due to the fact that the relevant measuring values for the calculation of the hardness are measured after removal of test force. Depth sensing indentation gives possibility to record simultaneously the acting force and corresponding penetration depth. In case of submicron dimensions this technique is called "nanoindentation" and is an important and effective experimental method. The instrumented nanoindentation testing provides a common method for measuring mechanical properties of materials on small scales. The indentations were carried out on Nanoindenter XP. A three-faced pyramidal Berkovich diamond indenter with semi-apical angle 65.3° and tip radius ~100 nm was used for investigations. The force-displacement raw data recorded in each test during DSI were first processed with a deduction of the previously calibrated machine compliance and respecting other effects (indenter tip bluntness, temperature drift, ...).

The hardness value measured by this method is called universal or microindentation hardness (STMMPPMP, 2006; DIN, 1997). The universal hardness value, HU (N/mm$^2$), is given by the ratio of the test force, $F$, to the area of the indentation under the applied test force. A typical curve from DSI data in Fig. 4 consists of three parts: loading, unloading and creep during dwell time at maximal load $F_{max}$. $h_{max}$ is corresponding indentation depth. $h_f$ then corresponds to final depth during unloading when the force $F$ drops to $F = 0$. It roughly expresses depth of residual impression which size on surface is optically measured to evaluate quantity of hardness in the traditional sense.

A revised linear analytical method was used in deriving unloading compliance $C_S$ and contact depth $h_C$ values, in which 50% of the unloading data were used for a power law curve fitting. Diamond indenter area function (which defines the relation of indent projected area $A_C$ and contact depth, $h_C$) has been independently calibrated as:

$$A_C = k_1(h_C)^2 + k_2 h_C \tag{1}$$

Therefore, following equations can be used in calculating hardness $H$ and Young's modulus $E$ values of the test samples:

$$H = \frac{F_{max}}{A_C} \tag{2}$$

$$E_r = \frac{\sqrt{\pi}}{2C_S\sqrt{A_C}} \tag{3}$$

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E} \tag{4}$$

where $E_r$ is reduced modulus, an intermediate parameter for the non-rigid inter-specimen contact effect. $E_i$ and $\nu_i$ are Young's modulus and Poisson's ratio for the (diamond) indenter, which can be taken as 1140 GPa and 0.07, respectively. $E$ and $\nu$ are then Young's modulus and Poisson's ratio for the test sample.

The available published data concerning to microhardness measurements are related to the methods applied in sintered powder compacts, e.g. (STMMPPMP, 2006; Oliver and Pharr, 1992; Bocchini et al., 2002; Bocchini et al., 2004, 2005; Molinari et al., 2001). In a matter of fact, there exists no exact