HARDNESS OF SINTERED PM MATERIALS WITH MICROGRADIENTS IN COMPOSITION

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Abstract
Particles of iron powder were electrolytically coated with a copper and subsequently with a hydrocarbon. Coated powder was compressed into cylindrical samples at 600 MPa and sintered at 1120°C. Using Microhardness Tester LECO LM-700AT, Vickers microhardness of samples has been investigated by indenting the unetched metallographic polished section. Test loadings were 10g, 25g, 50g, 100g, 200g, 300g, 500g and 1000g. Loading time was set on 10 seconds. It was found that there are different values of microhardness within a grain. Microhardness measurements taken at different points in the specimen enabled us to map the occurrence of various hardness values, which reflects a variety of the local microstructure of the examined material. At large loading, effective properties of a large volume of material are measured and the range of measured hardness narrows. ISE was observed.

Keywords: Iron powder, coating, powder metallurgy (PM), sintering, microhardness measurements, microstructure

1 Introduction
Iron-copper-carbon systems are very common materials for sintered components. Properly heat-treated parts made from these alloys possess higher mechanical properties than products sintered from a pure iron [1, 2, 3]. The importance of copper as an alloying element in the iron powder metallurgy is that the solid solution or precipitates cause the strengthening of resulting material and the copper enables sintering at the presence of liquid phase [4, 5]. The role of carbon in PM is threefold: (i) reduction of the internal and surface oxides introduced by powder particles, (ii) alloying, and (iii) improvement in densification of powder (carbon can act as a lubricant and binder) [2]. In order to distribute the alloying elements uniformly, iron powder particles are often coated with a proper layer containing those elements. The coating not only removes the segregation of elements during transport and processing of the powders, it also protects powders against further oxidation, improves compressibility, enhances dimensional stability upon sintering, and reduces harmful dustiness in the PM industry [2]. Indentation is frequently used to assess the mechanical properties of solids. Since the technique is based on the production of a small permanent indent on the surface by pressing a hard...
indenter into the solid, it is essentially non-destructive in contrast to the methods involving compression, extension or bending of macroscopic samples [6, 7]. The intention of the paper was to determine hardness values at various points of samples at different loads.

2 Experimental material and methods
Water-atomized iron powder, Höganäs ASC 100.29 grade, fraction 63 – 100 µm was used as a starting raw material. Particles were coated by Cu galvanostatically in a fluidised bed electrolyser. Fe/Cu particles were prepared under following conditions: composition of electrolyte: 0.06 M CuSO$_4$.5H$_2$O + 0.25 M C$_6$H$_5$Na$_3$.2H$_2$O; quantity of iron powder 30 g, period of electrolysis 30 min, current intensity 1 A, copper anode. Schematic sketch of fluidised bed electrolyser with its parameters are presented in [8]. Content of the copper was determined by method AAS. Fe/Cu particles were consequently coated by a layer of C$_n$H$_m$. Powder, prepared by pyrolysis of polyvinylalcohol in the non-reactive atmosphere at 663 K/35 min, was at first milled and then dissolved into xylene (C$_6$H$_4$(CH$_3$)$_2$). Fe/Cu powder was admixed to the resulting solution and xylene was evaporated while continuously stirred. Fe/3.3wt%Cu/C$_n$H$_m$ powder with 0.84wt%C was obtained. C$_n$H$_m$ coating was evenly distributed on the Fe/Cu particles. Details of production of the powders are presented in [2, 9, 10]. Coated powder was cold pressed in a cylindrical die with 10 mm diameter by 600 MPa press into final height around 10 mm. None lubricant was added to the powder. Zinc stearate was used as a die wall lubricant [11]. Pressed cylindrical samples were sintered in Marsch’s laboratory furnace at 1120°C for 1 hour in the reduction atmosphere 90% N$_2$ – 10%H$_2$. Powdered or compacted samples for metallographic examination were cross-sectioned, mounted, ground, and polished using well established practices [11, 12, 13]. The microstructure of samples was investigated with a light optical microscope (OLYMPUS GX71, Japan). Using microhardness tester LECO LM – 700AT, Vickers microhardness of samples has been investigated by Vickers diamond indenter.

3 Results and discussion

![Fig.1 Microgradient structure of sample pressed from coated powders Fe/3.3wt%Cu+C$_n$H$_m$ and sintered at 1120°C, nital etched](image)

Samples were sintered at 1120°C. The final microgradient structure of sintered samples, made more visible by nital etching, is shown in Fig. 1. Cores of the original iron particles do not contain any alloying particles (or only trace amounts of them) and therefore remain non-etched.
(bright). Etched areas (dark) are surrounding ones which contains solid solution of Fe-Cu, precipitates of Cu and pearlite. Microhardness of the sample has been investigated by Vickers diamond indenter using Microhardness Tester LECO LM-700AT with indentation loadings from 10g to 1000g (0.1N-10N). Different rates of indents were accomplished for each loading level. At the load 10g, there were 300 indents, 25g 300, 50g 150, 100g 150, 200g 100, 300g 90, 500g 20, 1000g 20 indents. Each individual indent was impressed into pre-defined place of sample, averages of the indent diagonals were determined and Vickers hardness HV was calculated by means of relation [14]

\[ HV = \frac{1.8544 \times P}{d^2} \]  

(1.)

where \( P \) is indentation load and \( d \) is diagonal’s average length. Size of indent is growing with increasing load and therefore the size of examined volume of material increases too. Local properties of small volumes of material are tested at low load [15], and in fact effective (“averaged”) properties of a large volume of material are tested at a large load. In addition, the “indentation size effect”, it means an increase in investigated hardness with decreasing indent size, takes place. Because the material with a quasi-continuous distribution of alloying elements with gradient in their concentration was investigated, local measurements (at low loading) provide us with a full spectrum of hardness, which reflects the composition and concentration of alloying elements in different points of sample where indents were realised. As the load increases, larger volumes of material are tested, in which composition and concentration of alloying elements vary from point to point. Result of the measurement is an “averaged” response given by volume of material, representing its effective hardness. Spectrum of measured hardness is narrowing. When the size of an indent becomes significantly larger than characteristic dimensions of non-homogeneities in the composition and in the concentration of alloying elements, results of measurement is an effective macroscopic hardness, which is in the case of macroscopically homogeneous sample practically independent of the position of indent, which results in a rather narrow range of measured values (Table 1).

<table>
<thead>
<tr>
<th>Loading strength</th>
<th>( P=0.10 ) N</th>
<th>( P=0.25 ) N</th>
<th>( P=0.50 ) N</th>
<th>( P=1.00 ) N</th>
<th>( P=2.00 ) N</th>
<th>( P=3.00 ) N</th>
<th>( P=5.00 ) N</th>
<th>( P=10.00 ) N</th>
</tr>
</thead>
<tbody>
<tr>
<td>arithmetic average [GPa]</td>
<td>2.429</td>
<td>1.986</td>
<td>2.019</td>
<td>1.690</td>
<td>1.591</td>
<td>1.426</td>
<td>1.186</td>
<td>1.328</td>
</tr>
<tr>
<td>standard deviation [GPa]</td>
<td>0.670</td>
<td>0.458</td>
<td>0.551</td>
<td>0.274</td>
<td>1.591</td>
<td>0.225</td>
<td>0.102</td>
<td>0.114</td>
</tr>
</tbody>
</table>

4 Conclusions
Iron powder particles were at first electrolytically coated with a layer of Cu and subsequently with a layer of \( \text{C}_n\text{H}_m \). Cylindrical samples from the coated powders were prepared by compacting under pressure of 600 MPa and by sintering at 1120°C. Sintered samples had microgradient structure where within individual grains there is occurring quasi-continuous
distribution of alloying elements with gradient in their concentration. Hardness measurements reflect the composition and concentration of alloying elements in various sample points at low loading. Hardness range is relatively wide. At large loading, effective (“averaged”) properties of large volume of material are measured and range of measured hardness narrows. “Indentation size effect” was observed, it means an increase in measured hardness with decreasing size of indent.

References

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