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On a discrepancy in modulus of elasticity as determined from separate resonance frequencies of a bar sintered from copper-coated iron powder

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Iron powder particles were coated with a copper, compacted into a bar shape, sintered and machined. Bars were caused to resonate longitudinally, transversely edgewise and flatwise. Three separate determinations of elastic modulus were made from the resonance frequencies of these modes. Three different values of elastic modulus were obtained for each of the bars. The nature of the differences indicates that the bars possess an inherent "layered" structure with stiffness graded transversely to the compaction direction. This supports a recently considered hypothesis about the behaviour of powders.

Keywords: Copper-coated iron powder; Sintering; Mechanical properties testing; Elastic behaviour; Theory

In conventional "ingot" metallurgy, the uniform distribution of strengthening elements in the base material is desirable. Any lack of homogeneity is considered detrimental. The situation is different in powder metallurgy. Properly designed differences in properties of the interior regions and the contacts between powder particles may lead to a synergetic effect that improves the overall characteristics. And many experimental tests have shown that a controlled and repeatable heterogeneity enhances the material properties of sintered steels.

Locally controlled distribution of elements can thus open the way to a new generation of more sophisticated powder metallurgy products. Therefore, a comprehensive programme to produce microscopically heterogeneous sintered steels using coated powders was initiated. This study represents a part of the programme.

This contribution has a twofold purpose. The first is to state the values of the effective elastic modulus as determined from separate resonance frequencies of bars sintered from copper-coated iron powders. The second aim is to demonstrate that the observed discrepancies in the modulus may be caused by a macroscopic heterogeneity that is inherent in the specimens studied. The assumed plate-like morphology of bars supports a recent model [1] for behaviour of copper-coated iron powders during die compaction and the early stages of liquid-phase sintering.

The production of samples is only briefly sketched here as the details have already been described elsewhere [2,3].

Water-atomized iron powder, Höganäs ASC 100.29 grade, fraction 63–180 μm, was used as a starting material. Iron particles were put into an aqueous electrolyte containing copper sulphate and sulphuric acid. The copper sulphate content in the electrolyte controlled the parameters of the final coat. After a cementation process was completed, the iron particle surfaces were almost entirely covered with copper for both of the copper concentrations used (3 and 8 wt.%).

Coated powders were blended with a lubricant (0.6 wt.%) and some also with 0.5 wt.% graphite to investigate the effect of carbon. Different kinds of test bars were cold compacted at 600 MPa and sintered in pusher furnaces at 1120 °C for 60 min. The atmosphere

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was N_2 or H_2 for samples with or without carbon, respectively. De-waxing conditions were 600 °C, 30 min, N_2.

It is well known that ordinary compacts from mixed iron and copper powders undergo enormous swelling upon sintering. Allowing for this swelling mechanism, the copper-coated iron powders were expected to restrain expansion. Regrettably, anomalous dimensional changes were also observed for the coated-powder compacts. The expected shrinkage occurred only along the direction of compaction, whereas compacts surprisingly swelled transversely to that direction [3].

The dynamic resonant method [4] was used for determining the elastic modulus. As the sintered compacts were not of the appropriate shape, they had to be machined into proper prisms. Depending on the dies used, some samples required only a slight grinding while others was ground heavily. The sizes of the resultant samples were about $97.5 \times 12 \times 8.5\ \mathrm{mm}^3$ for the slightly ground bars and about $88 \times 5.5 \times 3.5\ \mathrm{mm}^3$ for the heavily ground bars. Bar heights varied with the powder used.

Young's modulus is related to the longitudinal and flexural resonance vibrations. Rectangular bars may vibrate in flexural resonance both flatwise and edgewise. Resonance frequencies of all these modes were measured. Longitudinal resonance frequencies were measured by means of the frequency tester Erudite (C.N.S. Electronic Ltd., UK) (e.g. [5]) at the Institute of Rock Structure and Mechanics of ASCR in Prague. Flexural resonance frequencies were measured employing the apparatus Grindo Sonic MKS "Industrial" at the University of Vienna.

The disagreement in values of elastic modulus related to different types of vibration was found for every one of the bars. For each bar, the modulus related to flexural vibrations normal to the compaction direction was the lowest one. The relation between the two remaining moduli depended on whether the sample was ground slightly or heavily. For a slightly ground bar, the modulus related to longitudinal vibrations was higher than the modulus related to flexural vibrations parallel to the compaction direction (Table 1). For a heavily ground bar, the modulus related to longitudinal vibrations was slightly lower than the modulus related to flexural vibrations parallel to the compaction direction (Table 2).

A discrepancy in modulus as calculated from separate resonance frequencies prevents the evaluation of the modulus of elasticity of the bar material. However, calculated formal moduli and the relations between them may still be utilized to gain some insight into the morphology of the bars.

Table 1. Effective modulus of elasticity as determined from separate resonance frequencies of a slightly ground test bar

Bar material	Modulus (in GPa) related to vibrations		
	Longitudinal	Flexural parallel to compaction axis	Flexural normal to compaction axis
Fe + 3Cu + C	142.5 ± 0.1	137.3 ± 0.2	129.8 ± 0.2
Fe + 3Cu	135.8 ± 0.1	131.7 ± 0.2	124.7 ± 0.1
Fe + 8Cu + C	136.0 ± 0.1	131.2 ± 0.4	124.6 ± 0.2
Fe + 8Cu	119.3 ± 0.1	116.3 ± 0.3	110.1 ± 0.3

Table 2. Effective modulus of elasticity as determined from separate resonance frequencies of a heavily ground test bar

Bar material	Modulus (in GPa) related to vibrations		
	Longitudinal	Flexural parallel to compaction axis	Flexural normal to compaction axis
Fe + 3Cu + C	144.2 ± 0.2	144.4 ± 0.5	140.4 ± 0.7
Fe + 3Cu	135.8 ± 0.6	135.8 ± 0.4	129.8 ± 0.1
Fe + 8Cu + C	134.6 ± 0.7	135.1 ± 0.6	129.7 ± 0.6
Fe + 8Cu	122.6 ± 0.2	124.7 ± 0.9	119.4 ± 0.1

In general, failure to obtain an agreement in the elastic moduli evaluated from separate resonance frequencies may indicate a lack of macroscopic homogeneity in the tested specimen. Due to axial shrinking and lateral swelling, bars with macroscopically graded material properties may arise. The major grading is expected transversely to the compaction direction.

For theoretical considerations, it is convenient to idealize real sintered bars as continuous bars made of an effective continuum material. The problem considered may be idealized in such a way because the smallest dimensions of test bars are very large compared with the largest grains or pores. Also, only a large-scale heterogeneity is able to modify the macroscopic behaviour of a bar [4,6]. In the situation considered, the only relevant heterogeneity on the scale of bar dimensions is that due to the macroscopic grading of properties across the bar. Thus, studied sintered bars can be treated as continuous bars with effective-material properties constant along and varying across the bar, that is, as bars possessing the effective fibrous-like structure.

So, the bar is imagined to be a bundle of longitudinal fibres parallel to the longitudinal axis of a bar. The material properties of each fibre are constant along its length. The properties of individual fibres may differ from each other. Due to the problem of symmetry, fibres are arranged so that the bar possesses two longitudinal planes of symmetry perpendicular to each other.

The deformation behaviour of a slender fibrous bar resonating longitudinally is adequately described by a rule of mixtures appropriate for the equal-strain condition. This leads to the following expression for the effective Young's modulus [7]:

$$E_l = \frac{1}{HW} \int \int E(h, w)\, \mathrm{d}h\, \mathrm{d}w \tag{1}$$

$E(h, w)$ represents the effective-material Young's modulus that can vary only along the height and width of the bar, but not along its length. The h and w axes are both axes of symmetry of the bar cross-section, whose origins are at the centroid of the cross-section (Fig. 1). Integration runs over the area of a rectangular cross-section of the height H and width W.

The modulus related to flexural resonance frequencies is often termed the "effective flexural modulus". For a homogeneous bar, this equals the Young's modulus of bar material. For a doubly symmetrical bar with effective-material properties varying throughout the cross-section, the effective flexural modulus was previously found to be (e.g. [8]):