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Prediction of density in porous materials by x-ray techniques

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Abstract

Prediction of density in porous materials during mass production is one of the most important factors in industry. The existing methods for measuring density such as hydrostatic weighing and microscopical examinations have some limitations and inherent problems associated with them. Recently, processes based on radiography have emerged. This paper deals with the in situ prediction of density of porous materials using X-ray techniques. For this purpose, experiments were performed at various density levels of powder metallurgy compacts. In situ X-ray radiography was then carried out on samples. A correlation has been made between the film density of radiographic film and the surface density of the compacts. A mathematical relationship was derived between the absorbed x-ray, the film intensity and surface density. Comparison of values of Experimental data and mathematical relationship reveals discrepancies up to 3%.

Keywords: powder metallurgy, surface density, in situ radiography, film density

Introduction

Measuring the density of porous materials is one of the most important factors to consider in the production of parts made by powder metallurgy. Density is linked with many factors, including method of densification, shape and size of the part, particle size a shape of powder, its compressibility, quality and amount of any lubricants employed, and rate of pressing. Density can influence mechanical strength, hardness, electrical conductivity, and magnetic and gas permeability of PM compacts [1].

The existing methods for determining density and the character of its distribution hydrostatic weighing, infiltration with a phosphor, measurement of electrical conductivity, and microscopical examination, are labor and time-consuming [1], since they involve sectioning and infiltrating specimens, and necessitate using expensive equipment. Moreover, these methods cannot be employed for direct density measurements in the course of manufacture of parts. Therefore new methods concerned are based on nondestructive methods such as gamma radiography [1], ultrasonic tomography [2], X-ray computed tomography [3, 8], Eddy current measurement [5]. The use of nondestructive methods in medical application for measuring tablets density [3] and coral skeletal density [6, 7, 8] are progressed. However, using these methods in mass production of PM compacts is time-consuming and slow and needs expensive and complicated equipments.

In situ measurement of density of PM parts with the aim of being accurate and quick with X-ray radiography is used in this paper. This method is based on the fact that a radiation flux passing through a compact, which changes quantitatively depending on thickness and density. Therefore density of film is varied by affect of radiation change fall on the film.

The aim of this work is to determine a correlation between the film and compact densities. The data which have been collected from experiment are discussed below. The experimental data and theoretical equation are compared and evaluated.

Experimental Procedure

Iron powder Hoganas ASC100.29 was used in all experiments. Reproducibility in results is of essential factors to consider. Thus, all samples had the same thickness so that the extent of x-ray absorptions is identical. For this purpose, based on the compressibility of the iron powder, correct amount of pressure was estimated using the general relationships to an accuracy of ±0.1mm in height. Compression pressures were in the range of 90 – 480 Mpa.

In situ X-ray rapid radiography were done for the compacts using t=2 min, E=140Kv, I=4mA with type IX100 0.027mm Pb 10×24Cm (Fuji Film Envelopak), and were developed under identical conditions. Film densities were measured using a film densitometer, three times for each sample.

Results and Discussion

The aim of this work is to determine a correlation between the film (D) and compact densities. The experimental data of powder and film densities of ten iron compacts are shown in table 1.

Table 1 – The amount of compact and film density of Iron compacts.

Specimen no.	Film density (D)	Powder density (ρ) (gr/cm^3)	Pressure (Mpa)	Powder mass (gr)
1	1.94	6.5758	480	10.57
2	1.927	6.3816	436	10.26
3	2	6.1985	393	9.97
4	2.14	5.9548	349	9.57
5	2.24	5.773	305	9.28
6	2.27	5.581	262	8.97
7	2.4	5.259	218	8.45
8	2.585	4.91	175	7.90
9	2.95	4.37	131	7.15
10	3.09	4.044	87	6.51

Both analytical and empirical methods were investigated to determine the relationship between film and compact densities. Film density changes linearly with respect to compact density. This is due to the fact that highly porous materials emit more X-ray through it and therefore the absorbed energy would be less, leading to lighter film. There may still be sources

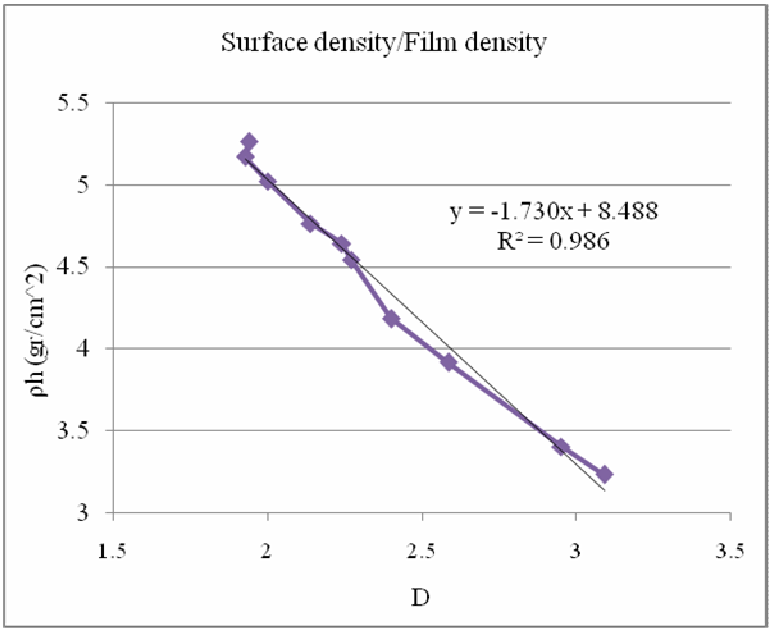

Figure 1. Film density versus surface density for iron compacts.

of error due to variations in height of the specimens, lubricant distribution, and press calibration. For this purpose, surface density was used instead of the compact density. This factor was determined by multiplying the compact density by the height of the specimen for each compact, as shown in figure 1.

There are still corrections to be made. When the pressure exerted is very low, green density tends to zero, suggesting that the absorption of the x-ray is negligible. Thus it would be difficult to quantify the film density in such circumstances. On the other hand, if the film density is zero, a reliable data can not be deduced from the radiography. However, the relationship between the film density and surface density can be estimated by the following model [1].

$$\rho h = \frac{1}{\mu} \, ln \frac{I_0}{I} \tag{1}$$

Where ρh is surface density, μ mass coefficient, I_0 and I are intensities of the incident and transmitted radiation.

It is suggested that surface density is related to the corresponding X-ray incident and transmitted the specimens, and also the amount of μ. This may be illustrated with a straight line; however it should pass through the origin of the diagram.

Using equation 1, when surface density is zero, the film density will be zero too. However in practice, it has its maximum value. This error can be corrected using this term substituting

$ln \frac{I_0}{I}$ =D and D'=1/D. The surface density can then be written as follows [9]:

$$\rho h = K \, \frac{1}{\mu \times 2.3026} \, D' \tag{2}$$

is correction factor in equation. K Where

Final results of this equation are shown in table 2:

Table 2 – Corrected experimental and theoretical results.

Specimen no.	**(ρ×h)$_{ex}$**	***D'***	**(ρ×h)$_{the}$**	***D*** **$_{the}$**
1	5.26064	0.51546	5.26064	0.5198406
2	5.169096	0.51894	5.10528	0.5044884
3	5.020785	0.5	4.9588	0.4900137
4	4.76384	0.46729	4.76384	0.4707483
5	4.641492	0.44643	4.6184	0.4563764
6	4.542934	0.44053	4.4648	0.4411981
7	4.186164	0.41667	4.2072	0.4157429
9	3.91818	0.38685	3.928	0.3881532
10	3.4086	0.33898	3.496	0.3454642
11	3.2352	0.32362	3.2352	0.3196927

Finally, the experimental and theoretical diagrams were plotted, as shown in figures 2 and 3.

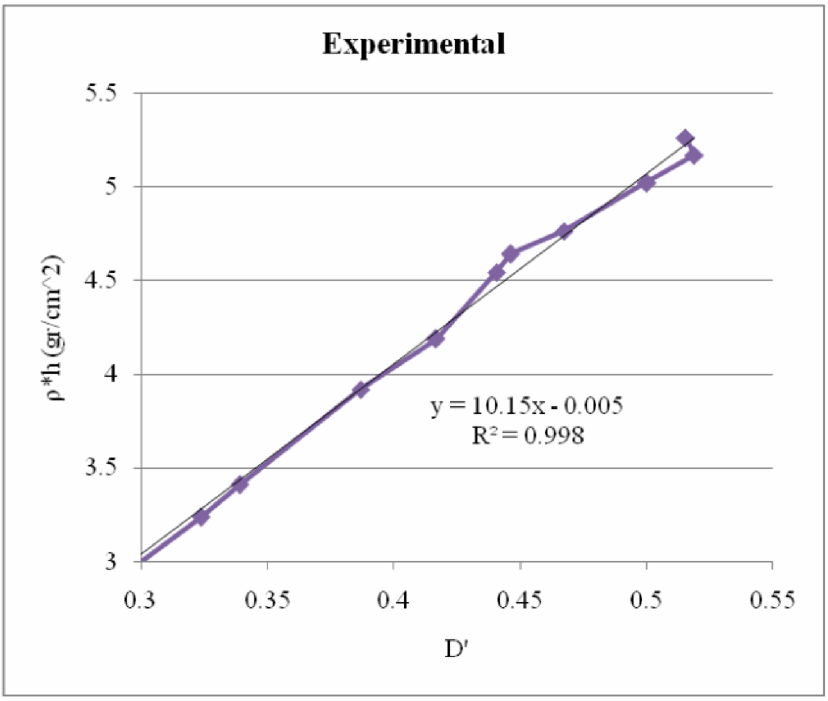

Figure 2. Surface density versus the film density based on experimental data.

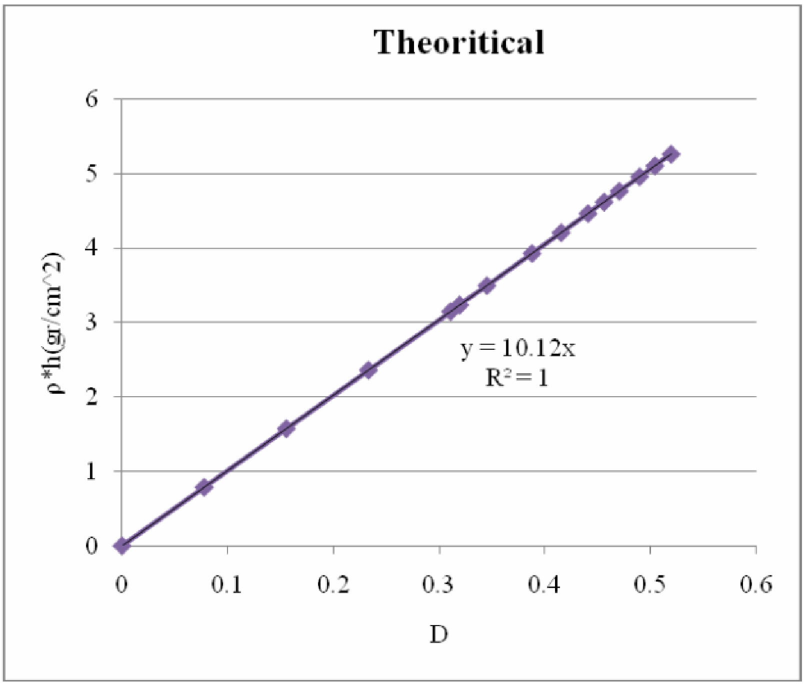

Figure 3. Surface density versus film density based on theoretical data.

Comparison of experimental and theoretical plots reveals that there is 0.5% error in the cross section of the straight line with the y axis, and an error of 3% in the slope of the line. This suggests that there is good correlation in using the suggested equation, to determine the density of Iron compacts.

Conclusion

In situ rapid radiography was performed to estimate the green density of iron compacts. It was shown that this technique can be applied to determine the green density of compacts. It was also found that a good correlation exists between the X-ray film density and its corresponding green density of Iron compacts. A correction method was employed to express the linear relationship between the variables. The deviation in the results between experimental and theoretical results reveals an error of 3%. The green density can therefore be estimated quickly using the proposed model, for porous compacts.

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