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Compressibility Studies on the Effect of Increase in Iron Content on Copper-Iron Powder Blends

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Abstract— Studies have been undertaken to experimentally determine the effect of increase in percentage of iron added to copper powder on the compressibility behavior and generating experimental data and then to analyze them systematically. The compositions selected for this purpose were Cu-0.0%Fe, Cu-1.96%Fe, Cu-2.91%Fe and Cu-4.76%Fe respectively. Compressibility assessments were carried out using 50g powder blends for each of the compositions. Powder height reduction was recorded systematically along with the applied pre-determined loads. Several graphs were plotted between various experimental and calculated parameters to investigate and test various compaction equations that are reported in the literature such as the Balshin, Heckel, Kawakita and Ge Rong-de including the ones arrived at in the present study. Several compressibility equations reported in the literature were tested for 50g powder/powder blends. It was found that the densification during compaction best corresponded to the proposed equations:

$$(\rho_f/\rho_{th}) = c_0 + c_1S + c_2S^2 + c_3S^3$$

$$(\rho_f/\rho_{th}) = b_0 + b_1\ln(H_0/H_c) + b_2[\ln(H_0/H_c)]^2$$

Where, ρ_f/ρ_{th} = fractional theoretical density or relative density S = applied stress, H_0 = Initial Height, H_c = Final Height The present investigation has reflected the compaction kinetics of copper powder blended with three different amounts of iron. Outcome of the present study can pave way for the successful production of P/M tools for many industrial applications subject to the condition that an appropriate blending is done and proper powder filling into the die is feasible.

Index Terms— P/M tools, Cu-Fe alloy, Compressibility, Compaction Equations.

I. INTRODUCTION

Powder Metallurgy is highly suited for manufacturing advanced composite materials as well as high stressed components which find extensive application in the automotive and consumer durables industry. The advantages of P/M technique include the ability to fabricate high quality complex products economically, minimization of machining and scrap losses, good surface finishes, maintaining close dimensional tolerances, allowance of a wide range of alloy systems, long term performance reliability in critical applications and many more. The major drawback of P/M is the porosity of the materials. In order to obtain a sound metallurgical structure and densities close to conventional materials elimination of voids to the maximum extent possible is essential. Therefore, it is necessary to know the manner in which the deformation of the pores takes place during loading.

P/M route basically has two processes. The primary P/M process includes powder production, powder blending, powder characterization, compaction and sintering. The pores in sintered P/M parts act as crack initiators during the application of these components in service and the crack initiation and its propagation would depend on the applied load and, therefore, in order to achieve full density, the pore closures become imperative [1-5]. Hence, the secondary deformation processes become essential. Kuhn [6] and Kahlow [7] have reported the material and the void response during powder preform forging. Thus, in comparison to wrought components, P/M parts exhibit good mechanical properties [8].

A. Theory of Compaction

The process of forming of metal powders into compacts of desired shape with sufficient strength to withstand ejection from tools and subsequent handling up to the completion of sintering, without breakage or damage is called Compaction. One of the most important methods for shaping metal powders is compaction in metal dies and even today accounts for the bulk of commercial production. Densification mechanism is mainly through deformation which may be both elastic and plastic in nature. Hence, the deformation or adhesion occurs between the individual particles or in between the particle conglomerates [9], a complex phenomenon. The compaction technique used in the present study is single compaction mainly used for manufacture of flat, thin parts. In the single action compaction process, the die, the bottom insert and the lower punch are stationary while the application of the pressure is done by the top punch.



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B. Compaction Equations

Compressibility data noted down includes noting down the distance through which the top punch has moved down by applying controlled pressure. With increasing pressure there arises larger areas of contact and as a result interlocking of neighboring particulates will take place. Smaller particles at the periphery squeeze into the voids thereby enhancing the density and strength. Thus, compressibility of a powder is related to the density attained at a preset compaction pressure. Efforts have been made for the development of empirical and theoretical compaction equations to describe the density-pressure relationships for the compaction of the powders. Out of more than twenty various compaction equations proposed, the most widely used equations are attributed to Balshin [10], Heckel [11] and Kawakita [12] respectively. One more widely applicable compaction equation with excellent accuracy and precision was developed by Ge Rong-de [13]. The purpose of the present study is to assess the influence of increase in the percentage of iron added to copper powder through testing of compaction kinetics.

II. EXPERIMENTAL DETAILS

Iron Powder (180µm) and Copper powder (37µm) were procured for conducting the experiment. The Cu-0.0%Fe, Cu-1.96%Fe, Cu-2.91%Fe and Cu-4.76%Fe compositions of the powders were taken for the preparation of powder mixture. Blending of powders was carried out in stainless steel pots on a pot mill separately for each composition. Powder to ball ratio was maintained at 1.1: 1 ratio and the blending operation was carried out for a period of 30 hours to obtain a uniform homogeneous mixture. The measure of the powder ability to deform under the applied pressure is termed as compressibility and is described by pressure-density relationships. In order to plot this curve, pre - weighed powder (50g) was taken in the die, the powder was subjected to different loads and the instantaneous height of the compact was calculated with the help of a dial gauge arrangement. The plot of density versus compaction pressure thus obtained was used for subsequent compaction. With an increase in compaction pressure, the compaction density is also expected to increase. Pressure has no effect after certain level of densification.

III. RESULTS AND DISCUSSION

This section deals with various plots drawn to correlate Log(Stress) against Log(H₀/H_c), Fractional Theoretical Density vs True Height Strain, Fractional Theoretical Density vs Stress, 1/D vs ln(P) and other different logarithmic plots so as to assess the true behavior of compactibility of copper powder and copper powder blends with iron.

Log (Stress) vs Log(H₀/H_c)

Figure 1 establishes the true relationship between the applied stress 'S' and the true compressibility (H₀/H_c). In order to achieve the above it becomes mandatory to plot between log (stress) and log(H₀/H_c) for all the systems investigated. It is observed from this figure that the neat straight lines represent the true behavior of the variation of log(stress) with log(H₀/H_c). Since all the plots representing the above compositions are straight lines, they can be best expressed mathematically as :

$$\log(S) = q\log(H_0/H_c) + \log(r) \dots\dots\dots(1)$$

where, 'q' and 'r' are empirically determined constants which depend upon compositions of the systems. However, the above expression can be expressed as a power law equation of the form : $S = r(H_0/H_c)^q$

Once again the values of regression coefficients are close to unity and, therefore, the above is the best fit relationship. The constants 'q' and 'r' are listed in Table 1.

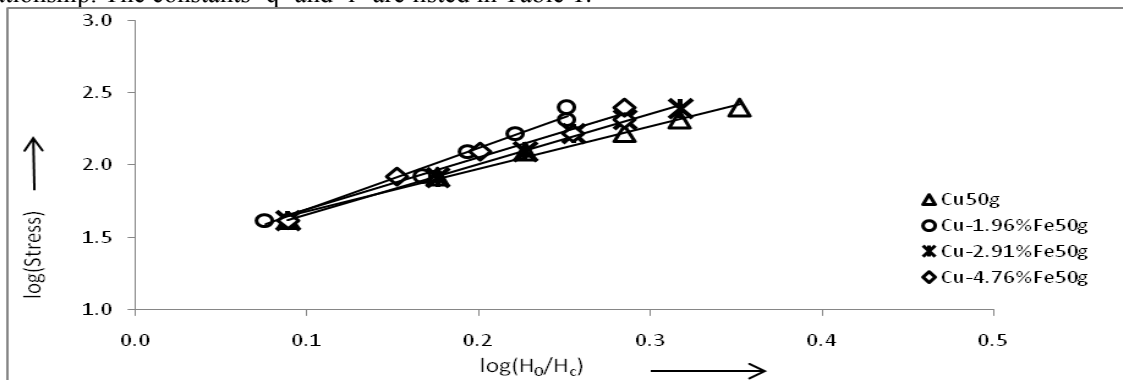


Fig 1 Plots Drawn Between Log (Stress) and Log (H₀/H_c) During the Compaction of Cu Powder With and Without Addition of Iron Powder of 180µm



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TABLE I The Coefficients and Intercepts of the Exponential Equation of the Form $\log(S) = q\log(H_0/H_c) + \log(r)$ for Cu and Cu-Fe Alloys During Compaction

Composition	Powder Weight	Coefficients		Regression Coefficient
		q	r	R ²
Pure Copper	50g	2.934	1.385	0.991
Cu-1.96%Fe	50g	4.279	1.264	0.979
Cu-2.91%Fe	50g	3.485	1.309	0.997
Cu-4.76%Fe	50g	3.595	1.333	0.980

S = Stress ; H₀ = Initial Height; ; H_c = Final Height

Fractional Theoretical Density and the True Height Strain

Figure 2 is drawn between fractional theoretical density (ρ_f/ρ_{th}) and the true height strain $[\ln(H_0/H_c)]$. All the curves are found to be similar in nature and, thus, they were found to conform to a second order polynomial of the form :

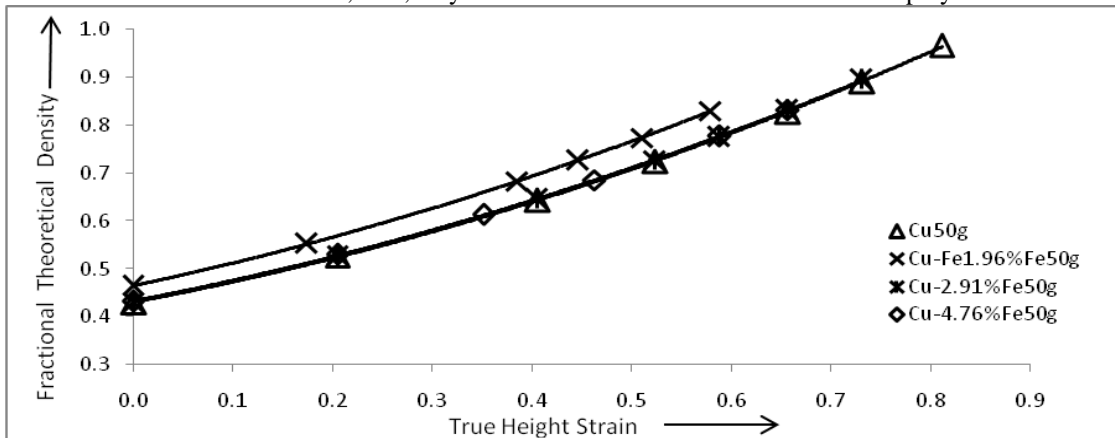


Fig 2 Plots Drawn Between Fractional Theoretical Density and the True Height Strain During the Compaction of Cu Powder With and Without Addition of Iron Powder of 180µm

$$(\rho_f/\rho_{th}) = b_0 + b_1 \ln(H_0/H_c) + b_2 [\ln(H_0/H_c)]^2 \dots\dots\dots(2)$$

where, 'b₀', 'b₁' and 'b₂' are found to be empirically determined constants and they do depend upon the compositions of the systems. These constants along with the regression coefficient values are given in Table 2. All these constants are found to be positive but, since 'b₀' is not multiplied by true height strain, it does not contribute to densification. However, within the limits of the test the constants 'b₁' and 'b₂' contributed to densification. Since the values of all regression coefficient are found to be almost unity and therefore the attained relationship is most appropriate.

TABLE II The Coefficients of the Second Order Polynomial of the Form $(\rho_f/\rho_{th}) = b_0 + b_1 \ln(H_0/H_c) + b_2 \ln(H_0/H_c)^2$ for Cu and Cu-Fe alloys During Compaction

Composition	Powder Weight	Coefficients			Regression Coefficient
		b ₀	b ₁	b ₂	R ²
Pure Copper	50g	0.430	0.392	0.327	0.999
98.04%Cu-1.96%Fe	50g	0.465	0.441	0.321	1.000
97.09%Cu-2.91%Fe	50g	0.431	0.398	0.318	0.999
95.24%Cu-4.76%Fe	50g	0.432	0.410	0.302	1.000

(ρ_f/ρ_{th}) = Fractional Theoretical Density ; H₀ = Initial Height ; H_c = Final Height

Relationship between Relative Density and the Applied Stress

Figure 3 is drawn between relative density and the applied stress exhibiting the influence of iron powder of 180µm addition in copper powder during compaction. The curves shown in this figure are found to be characteristically similar to each other, and, therefore, they can be expressed by a quite similar mathematical expression. Curve fitting has resulted in the existence of a third order polynomial between relative density and the applied stress of the form :

$$(\rho_f/\rho_{th}) = c_0 + c_1 S + c_2 S^2 + c_3 S^3 \dots\dots\dots(3)$$



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Where c_0 , c_1 , c_2 and c_3 are empirically determined constants and are found to depend upon the compositions of the systems. These constants are tabulated in Table 3 along with the values of the regression coefficients. The constant c_0 represents the initial loose density of the copper powder and copper-iron powder blends and, therefore, this constant does not contribute to densification. The constants c_2 and c_3 are quite small, but, are multiplied by the square and cube terms of the stress. Thus they contribute to plateauing of the curves in the final stages of compaction.

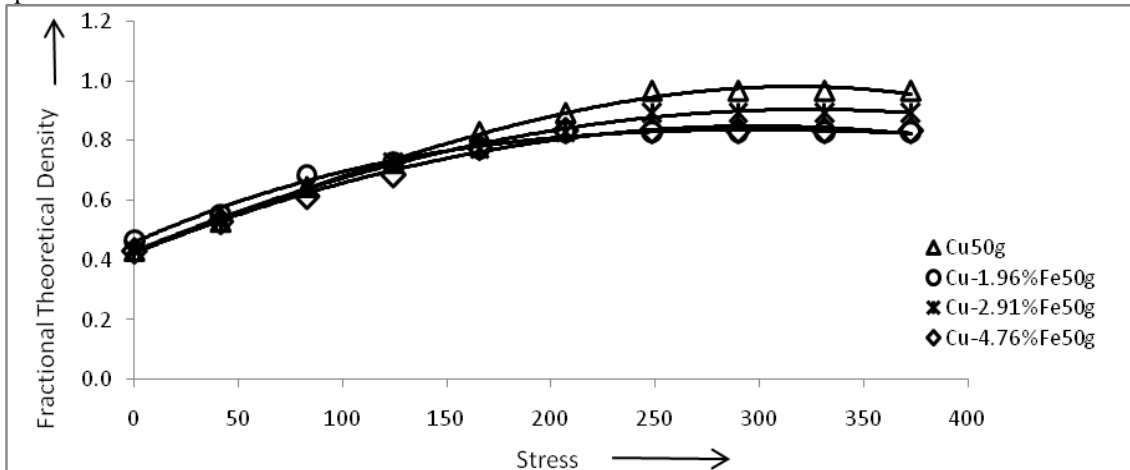


Fig 3 Plots Drawn between Fractional Theoretical Density and the Applied Stress during the Compaction of Cu Powder With and Without Addition of Iron Powder of $180\mu\text{m}$

Though the constant c_1 is small, it is multiplied linearly by the stress and is positive, and, therefore it contributes to densification during compaction. Further, the values of the regression coefficient are close to unity, and hence the relationship arrived at stands most justified.

TABLE III The Coefficients of the Third Order Polynomial of the Form $(\rho_f/\rho_{th}) = c_0 + c_1S + c_2S^2 + c_3S^3$ for Cu and Cu-Fe Alloys During Compaction

Composition	Powder Weight	Coefficients				Regression Coefficient R^2
		c_0	c_1	c_2	c_3	
Pure Copper	50g	0.424	0.002	-8E-08	-9E-09	0.997
Cu-1.96%Fe	50g	0.457	0.003	-8E-06	7E-09	0.991
Cu-2.91%Fe	50g	0.427	0.002	-4E-06	-6E-10	0.996
Cu-4.76%Fe	50g	0.425	0.002	-4E-06	-8E-10	0.991

(ρ_f/ρ_{th}) = Fractional Theoretical Density ; S = Stress

Testing of Existing Compaction Equations

A. Balshin Equation

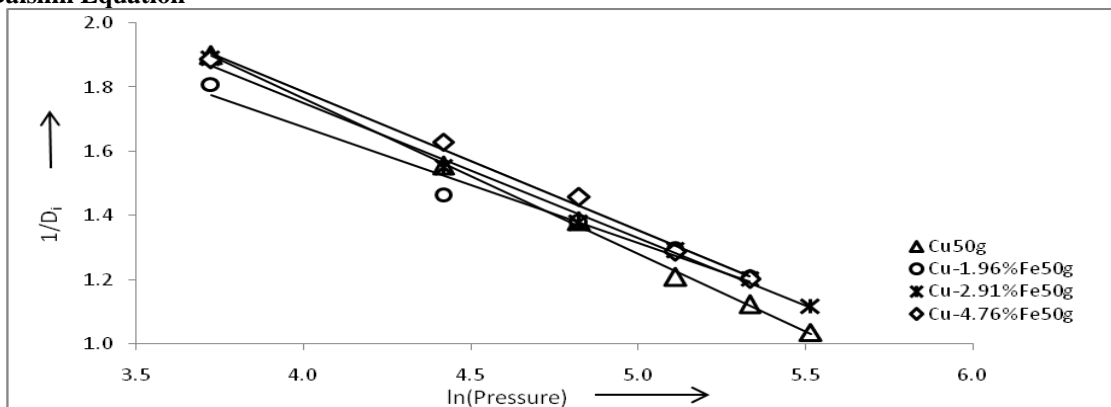


Fig 4 Plots Drawn Between $1/D_i$ and the $\ln(\text{Pressure})$ During the Compaction of Powder With and Without Addition of Iron Powder of $180\mu\text{m}$

Balshin proposed that a plot between inverse of achieved density (D_i^{-1}) with natural logarithm of applied stress (Pressure) would be a straight line, i.e. $D_i^{-1} \propto \ln(S)$ or

$$1/D_i = M \ln(S) + C \dots\dots\dots(4)$$

Figure 4 has been drawn between inverse of density and $\ln(S)$. This figure clearly shows that almost all the respective data points corresponded to an independent line exhibiting a linear behavior. The corresponding constants, namely the slope (M) and the intercept (C) do depend on the compositions of the systems and are tabulated in Table 4 along with the values of the regression coefficients. The Balshin equation was most suited in three cases as the values of regression coefficients were close to unity, whereas it was 0.976 in the case of Cu-1.96%Fe blend and hence the validity in this case was just satisfactory.

TABLE IV The Coefficients and Intercepts of the Equation of the Form $1/(\rho_f/\rho_{th}) = M \ln(S) + C$ for Cu and Cu-Fe Alloys During Compaction

Composition	Powder Weight	Coefficients		Regression Coefficient
		M	C	R ²
Pure Copper	50g	-0.483	3.695	0.998
Cu-1.96%Fe	50g	-0.361	3.123	0.976
Cu-2.91%Fe	50g	-0.422	3.438	0.994
Cu-4.76%Fe	50g	-0.431	3.511	0.993

B. Heckel equation

Heckel compaction equation relates between $\ln(1/(1 - D_i))$ and the applied pressure (S). He suggested that the above plots must be linear. Therefore, plots have been drawn between $\ln(1/(1 - D_i))$ and the applied pressure (S) from the experimental data generated for all the copper powder/ copper-iron blends. These plots are shown in figure 5. It is observed that all the plots in this figure are straight lines and the slopes and the intercepts of these lines do depend upon the blend compositions.

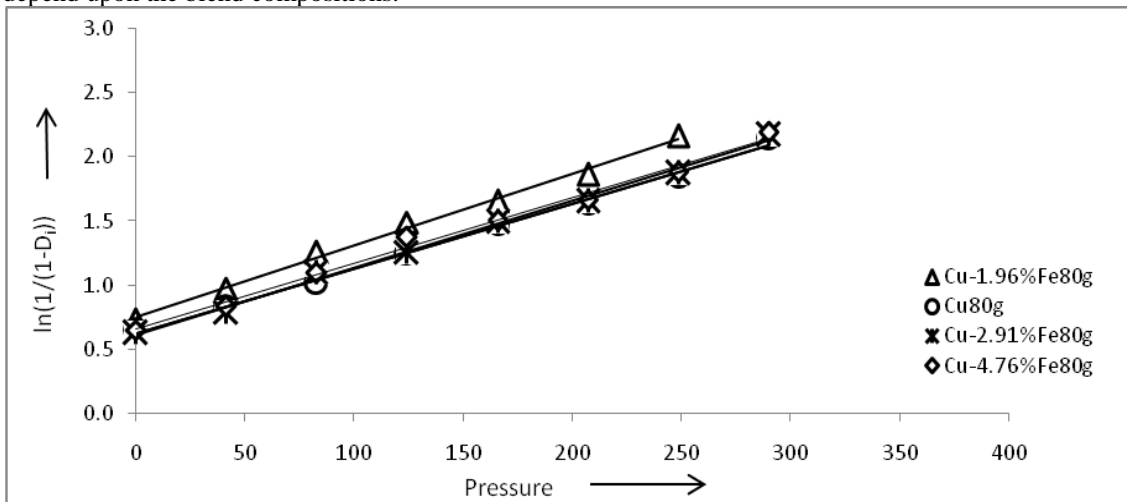


Fig 5 Plots Drawn Between $\ln(1/(1-D_i))$ and the Applied Stress During the Compaction of Cu Powder With and Without Addition of Iron Powder of 180 μ m

TABLE V The Coefficients and Intercepts of the Equation of the Form $\ln(1/(1 - D_i)) = M_1S + C_1$ for Cu and Cu-Fe alloys During Compaction

Composition	Powder Weight	Coefficients		Regression Coefficient
		M ₁	C ₁	R ²
Pure Copper	50g	0.007	0.490	0.973
Cu-1.96%Fe	50g	0.005	0.622	0.989
Cu-2.91%Fe	50g	0.006	0.499	0.983
Cu-4.76%Fe	50g	0.005	0.508	0.985

(ρ_f/ρ_{th}) = Fractional Theoretical Density ; S = Stress

These lines corresponded to a general linear equation of the form :

$$\ln(1/(1 - D_i)) = M_1S + C_1 \dots\dots\dots(5)$$



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Where M_1 and C_1 are constants and these constants were empirically determined and are found to depend upon the Compositions of the systems. Further, these constants along with the values of regression coefficients are tabulated in Table 5. Since the values of regression coefficients have deviations of 0.027, 0.011, 0.017 and 0.015 from unity for the above systems, it can be argued that the Heckel's equation's validity is considered satisfactory.

C. Kawakita Equation

Kawakita equation shows the relationship between $(D_i/(D_i-D_0))$ and inverse of applied stress. According to him, the plots obtained must be linear. The plots of (D_i/D_i-D_0) vs $(1/S)$ have been plotted in figure 6 for all the systems mentioned earlier. Clearly, all the plots in figure 6 are straight lines. The intercepts and slopes of these lines depend upon the Cu-Fe compositions. The general linear equation corresponding to these lines are of the form :

$$(D_i/(D_i - D_0)) = M_2(1/S) + C_2 \dots\dots\dots(6)$$

where M_2 and C_2 are constants and are empirically determined.

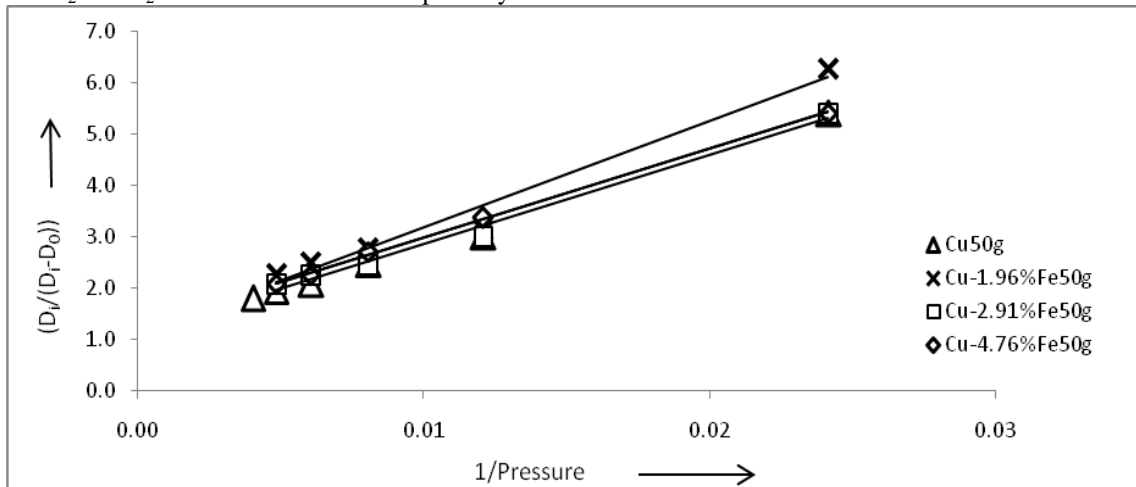


Fig 6 Plots Drawn Between $(D_i/(D_i-D_0))$ and $(1/P)$ During the Compaction of Cu Powder With and Without Addition of Iron Powder of $180\mu\text{m}$

TABLE VI The Coefficients and Intercepts of the Equation of the Form $(D_i/(D_i - D_0)) = M_2(1/S) + C_2$ for Cu and Cu-Fe alloys During Compaction

Composition	Powder Weight	Coefficients		Regression Coefficient
		M_2	C_2	R^2
Pure Copper	50g	178.8	1.013	0.995
Cu-1.96%Fe	50g	207.2	1.110	0.971
Cu-2.91%Fe	50g	173.3	1.126	0.989
Cu-4.76%Fe	50g	172.5	1.260	0.999

D_0 = Initial Fractional Theoretical Density ;
 D_i = Fractional Theoretical Density at applied Stress;
 S = Stress

These constants along with regression coefficients are tabulated in Table 6. Kawakita equation is found most valid for Cu-0.0%Fe, Cu-2.91%Fe and Cu-4.76%Fe blends as the values of regression coefficients are close to unity. However, its value is 0.971 only for Cu-1.96%Fe blend and hence the validity of the equation is considered as satisfactory only.

D. Ge Rong-de Equation

Figure 7 is plotted between $\text{Log}[\ln(1-D_i)/(1-D_0)]$ and logarithm of applied pressure for all the systems investigated. Ge Rong-de suggested that such plots must be straight lines. Thus, the plots drawn with the above parameters conformed to a general linear equation of the form :

$$\text{Log}[\ln(1-D_i)/(1-D_0)] = M_3\text{log}(S) + C_3 \dots\dots\dots(7)$$

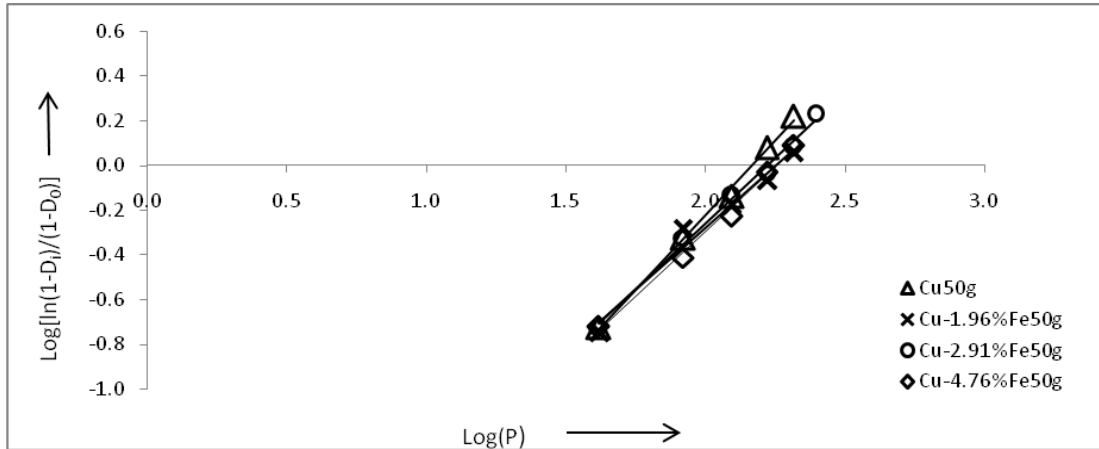


Fig 7 Plots Drawn Between and the Log $[\ln(1-D)/(1-D_0)]$ and Log(P) During the Compaction of Cu Powder With and Without Addition of Iron Powder of $180\mu\text{m}$

Where, M_3 and C_3 are empirically determined constants and are found to depend on the compositions of the systems. The values of the regression coefficients (R^2) and these constants are given in Table 7. Except for Cu-1.96%Fe blend ($R^2 = 0.972$) and for all the other three systems investigated conformed very well with Ge Rong-de equation as their values of the regression coefficients were found to be very much close to unity and hence the validity of the equation stands the test of trial extremely well.

TABLE VII The Coefficients and Intercepts of the Equation of the Form $\log [\ln (1-D_i)/(1-D_0)] = M_3\log(S) + C_3$ for Cu and Cu-Fe Alloys During Compaction

Composition	Powder Weight	Coefficients		Regression Coefficient
		M_3	C_3	R^2
Pure Copper	50g	1.339	-2.902	0.995
Cu-1.96%Fe	50g	1.103	-2.485	0.972
Cu-2.91%Fe	50g	1.170	-2.600	0.993
Cu-4.76%Fe	50g	1.159	-2.619	0.993

D_0 = Initial Fractional Theoretical Density ;

D_1 = Fractional Theoretical Density at applied Stress ;

S = Stress

IV. CONCLUSION

Based upon the experimental data and various calculated parameters with series of plots drawn and their critical analysis has revealed the following major findings of the present investigation:

1. It has been established that the applied stress followed a Power Law equation with the compressibility factor and the expression was of the form:

$$S = r(H_0/H_c)^q$$

where, q and r are empirically determined constants. Since regression coefficients for all the systems were quite close to unity, with little more deviation for Cu-1.96%Fe system.

2. The densification pattern during compaction followed a second order polynomial with very close values of regression coefficients in the vicinity of unity or unity proves to be the best fit where b_0 , b_1 and b_2 were empirically determined constants and depends upon the compositions of the systems and this relation is the best fit.

3. Similarly the relationship between fractional theoretical density and the applied stress conformed to a third order polynomial of the form :

$$(\rho_f/\rho_{th}) = c_0 + c_1S + c_2S^2 + c_3S^3$$

where c_0 , c_1 , c_2 and c_3 are empirically determined constants and the same were functions of compositions of the systems. Once again the values of R^2 were beyond 0.99 and therefore this has been the best fit.



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4. While testing the validity of Balshin, Heckel, Kawakita and Ge Rong-de compaction equations, it has been established that except in the case of Cu-1.96%Fe, for the remaining 3 systems, they were the best fits as in all cases, the values of regression coefficients has been beyond 0.99.
5. In summary, systems investigated and the experimental data obtained with their calculated parameters and their analysis has established that the already existing compaction equations like Balshin, Heckel, Kawakita and Ge Rong-de could be utilized successfully but the two expressions proposed in this present investigation exhibited much better fits with lesser complexities and the same can be utilized in future investigations when compactibility becomes important.

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