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Literature References

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COMPRESSIBILITY OF METAL POWDERS

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Abstract

The mathematical relationship between net pressure (expanded directly on densification) and relative density of die pressed compacts has been established. The dependence relates such powder characteristics as apparent density, compactibility (compressibility), sliding and friction coefficients against the material of a die. The proposed description of a compacting process is verified for copper alloy and tungsten carbide powders. The relationships, consistent with experiment, may serve as a basis for more precise design and control of powder preparation and compacting processes.

Introduction

The main drawback of a die pressing is the nonuniform density distribution throughout the compact. The friction between powder and die's walls as well as between all individual particles hinders a diffusion of pressure during compaction and, in effect, variations in density. On the other hand, high quality of a final product requires the density in a compact to be as uniform as possible. The factors affecting the density distribution are: the type of the compacting technique, the type of tools used, and the properties of the powder. Therefore, for a given tool design from a given material, the density distribution throughout the compact depends only on powder properties.

Among the most important powder parameters are the following: flowability of a loose powder, apparent (green) density, friction coefficient between powder and die's walls, and compactibility (compressibility) coefficient.

The compactibility and apparent density are used to estimate the efficiency of a compacting process which is restricted by the time interval needed to fill up a die with a powder in an automated process. The coefficient of friction is of crucial importance to the technical side of the compacting process. It depends on the powder properties, the material of the die, and the quality of die's surfaces. Its magnitude characterizes the powder's capability for uniform densification along the height of the compact. The compactibility coefficient is assumed to depend on powder properties (such as interparticle friction) and may be treated as a measure of powder's capability to be compacted.

At the present time there are commonly used methods for determination of the apparent density and the flowability of powders but no reliable method exists for the determination of the friction and compactibility coefficients.

Friction Coefficient and Slide Coefficient

Many studies have been carried out to express in a mathematical form the distribution of the pressing forces in dies as a function of related properties and relationships during compacting. The most interesting formula derived for one-end pressing of cylindrical samples (Fig. 1) is given by Ballhausen [1]. It can be generalized for arbitrary samples of constant cross-section as:

$$\frac{p_c}{p_d} = e^{\left(\mu \tan \phi \frac{SH}{F}\right)}$$

Here, p_c is the pressure applied to the top punch, p_d is the pressure transmitted to the bottom punch, μ is the friction coefficient, ϕ is the angle of the pressure transmission from the top to the die's wall, S is the perimeter of the cross-section, H is the height, and F is the area of the cross-section.

The magnitudes of p_c, p_d, S, H, and F above can be easily measured. In order to determine the friction coefficient μ it is necessary to correctly estimate the angle ϕ. As yet, no reliable method has been proposed for the determination of ϕ and, consequently, an accurate friction coefficient μ cannot be obtained. Based on the relation above, the magnitude of the friction coefficient can vary over a wide range.

A similar approach is used by Gasiorek and his co-workers [2,3] who introduced the following empirically established relationship,

$$\frac{p_d}{p_c} = \eta^{\frac{SH}{4F}}$$

Here, η is the coefficient of sliding friction or, in short, slide coefficient. The slide coefficient characterizes interactions between the powder and the die's walls. For a given material and surface condition of a die, the coefficient is a constant and describes the powder's ability to be uniformly densified during compaction. Its numerical value can vary in the range $0<\eta<1$

The above relation allows one to determine the slide coefficient from experiment. Combining the above equations one obtains the relations between the slide coefficient and the friction coefficient,

$$\eta = e^{-4\mu \tan \phi}$$

Numerous experiments [2,3] with various cylindrical samples pressed from ferrite powders subjected to a wide range of technically applicable pressures have shown that the slide coefficient is indeed a constant. Using the relations above, we can rewrite them for local pressure p_h at a distance h from the pressing punch,

$$p_h = p_c \eta^{\frac{Sh}{4F}}$$

This relation allows one to predict the pressure at any particular cross-section of the pressed compact once the slide coefficient is known.

The non-uniform pressure distribution creates a non-homogeneous density distribution in the compact along the pressing direction. Despite the great technical importance of the density-pressure dependence, not much attention has been devoted to that aspect with relatively few papers reporting results of mainly direct density measurements [4]. Such measurements are subjected to considerable errors that preclude any generalized description of the phenomena accompanying the compacting process.

It has been determined [2,3] that the density distribution along the height of the compact is approximately linear. Thus, the local density measured at the half height of the compact, $h=H/2$, is equal to the average apparent density of the whole compact. The pressure p_r at that height can be found from the above relation for local pressure,

$$p_r = p_c \eta^{\frac{SH}{8F}}$$

The introduction of the net pressure concept allows one to determine the compaction characteristic independent from the $SH/4F$ quantity. In addition, it could be shown that the above compaction characteristic for die pressing is the same as the compaction characteristic obtained for isostatic pressing process.

Compactibility

Many mathematical relationships have been established between the nominal pressure applied to the upper punch and the average density of the compact produced. Those relationships are important from practical point of view. However, since only nominal or average values of pressure and density are used in those equations [5,6,7,8,9,10] it is difficult and often impossible to establish a fundamental general theory. An extensive experimental investigation [2,3] has indicated a logarithmic functional dependence between the net pressure and the density:

$$|log\,|log\,\rho_{ra}|| = f\,(log\,p_r)$$

where ρ_{ra} is an average relative density of a given sample. Furthermore, the function f has been shown to be linear,

$$|log\,|log\,\rho_{ra}|| = \alpha\,log\left(\frac{p_r + p_o}{p_o}\right) + |log\,|log\,\rho_{rp}||$$

where α is the compactibility coefficient, p_o is the gravitational pressure of the powder, and ρ_{rp} is the relative density of a bulk powder.

The above expression can be transformed to,

$$\rho_{ra} = \rho_{rp}^{\left(\frac{p_o}{p_o+p_r}\right)^{\alpha}}$$

Replacing the reduced pressure p_r with the local pressure p_h, we get an equation for the density distribution along the height of a given sample in the form:

$$\rho_{rh} = \rho_{rp}^{\left(\frac{p_o}{p_o+p_c\,\eta^{Sh/4F}}\right)^{\alpha}}$$

The relations and concepts for determination of powders' compactibility have shown very good correlation with experimental data for ferrites and few other powders of metal oxides [2,3].

The main purpose of this work has been to determine the application of the above concepts to powders other than metal oxides.

Experimental Procedures

Two different types of powders have been selected for compactibility determination:

1. Tungsten carbide, very hard and fragile material. Due to the lack of plastic deformation range, its properties are similar to properties of ceramic materials based on oxides and silicates. Dry pressing is the most widely used process in forming parts. The hard grains cannot be deformed during pressing and consolidation by van der Waals attractive forces and electrostatic forces are weak. For that reason powders prepared for pressing contain 2% (by weight) of wax as a binder. The pressed parts have the same relative density range of .56 to .64 as oxide and silicate compacts. To reache the above density range, a net pressing pressure in the range 1000 to 3000 kG/cm^2 is required.

2. Mixture of a bronze (80%) and iron (20%) powders pressed in the plastic deformation range. Relative density of the pressed compacts is in such cases usually much higher than for oxides and silicates. Plastic deformation of the powder grains during pressing results in relatively high mechanical strength of compacts without binders. For more uniform density distribution along the pressing direction, a zinc stearate (.2% by weight) has been added as a lubricant.

Several tests have been performed on the above powders. The testing equipment, the Powder Testing Center Model PTC-01, is a commercial testing instrument based on the principles described above and manufactured by the KZK Powder Technologies Corp in Cleveland, Ohio.

Results and Discussion

As seen in the expressions above, the geometrical details enter those equations through a non-dimensional expression Sh/4F. For simplicity we introduce it as a separate parameter defined as Ga=Sh/4F. Figure 2 shows the p_h as a function of Ga and Figure 3 the apparent (local) density as a function of Ga for a tungsten carbide compact pressed to an average relative density ρ_{ra}=.595. With the slide coefficient η=.57 and Ga_{max}=1.2, the pressure applied to the top punch is 1620 kG/cm^2 and the pressure transmitted to the bottom punch is only 800 kG/cm^2, with the difference taken by friction. This difference in internal pressure generates a variable density along the pressing direction in the compact equal to .4 g/cc (see Fig. 3).

Figure 4 shows the ratio of the pressure at the bottom punch and at the pressing punch, p_d/p_c, as a function of Ga_{max} and Figure 5 illustrates local relative density for a compact with Ga_{max}=1.2 pressed from a mixture of bronze and iron powders with an average relative density of .73. The compacting characteristics of this powder are much better than those for tungsten carbide. With relatively high slide coefficient of .75, the density distribution along the height of the compact is much more uniform than that for tungsten carbide. The loss of pressure due to the powder/die's walls friction and, consequently, ejection force during removal of the compact from the die are 18% lower. Numerous experiments with ceramic and metallic powders [11,12] have indicated that slide coefficient of metallic powders against steel die wall is usually higher than for ceramic powders.

The above relationships were investigated for very wide average density range of compacts from both powders. Based on those tests, the dependence of the slide coefficient on average relative density has been checked. As seen on Figure 6, the slide coefficient is a constant independent from average density variations for both investigated powders. The maximum spread of the experimental points is on the order of .04, in the vicinity of the experimental accuracy.

Figures 7 and 8 show compaction characteristics for the tungsten carbide and bronze based powders, respectively. The relationship between compactibility coefficient and average density of compacts from those powders is shown on Figure 9. The compactibility coefficient for the tungsten carbide powder is constant in the relative density range from .45 to .56. In the range from .56 to .635 the compactibility coefficient slightly increases. This change may be related to the change of the densification mechanism caused by higher concentration of the binder on the surface of powder grains.

In the other case involving the bronze based powder, the compactibility coefficient changes linearly with the density. Here, the powder has been pressed beyond its proportional elastic limit. There is a great probability that the continuous change in the densification mechanism occurs along the height of a compact.

Conclusions

The results presented in this paper have shown very good conformity with mathematical relationships shown earlier. It has been demonstrated that the capabilities of tungsten carbide and the mixture of bronze and iron powders for uniform densification during pressing may be fully characterized by means of the slide coefficient. This enables one to optimize powders, that is to choose the most efficient binders and lubricants. The detailed knowledge of the local pressure and density simplifies a powder compacting process design.

The compactibility coefficient as defined in this paper has been shown to be a constant for powders of brittle materials which do not have the yield point such as oxides, silicates, carbides, and nitrides. A small change of the compactibility coefficient has been observed in powders containing aggregates, harder conglomerates, or larger amounts of binders. For metallic powders, the compactibility coefficient most likely will never be constant in a practical density range. The densification mechanism is affected by the powder grains deformations above the proportional elastic limit. Therefore, the local density variations as derived in the chapters above will have to be evaluated for a variable compactibility coefficient. For all practical purposes in a narrow range of density variations the compactibility coefficient can be assumed to be constant.

Acknowledgment

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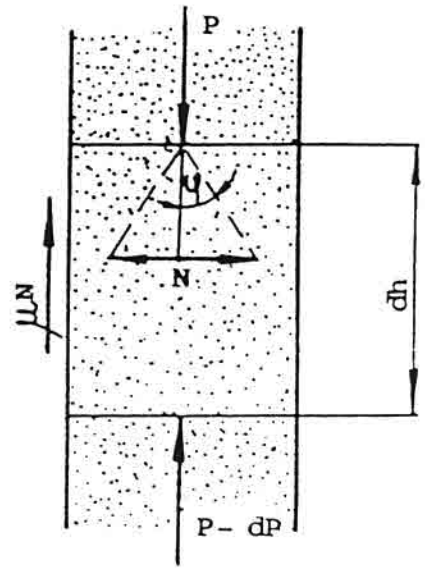


Figure 1. Distribution of forces acting on a selected element of a green compact.

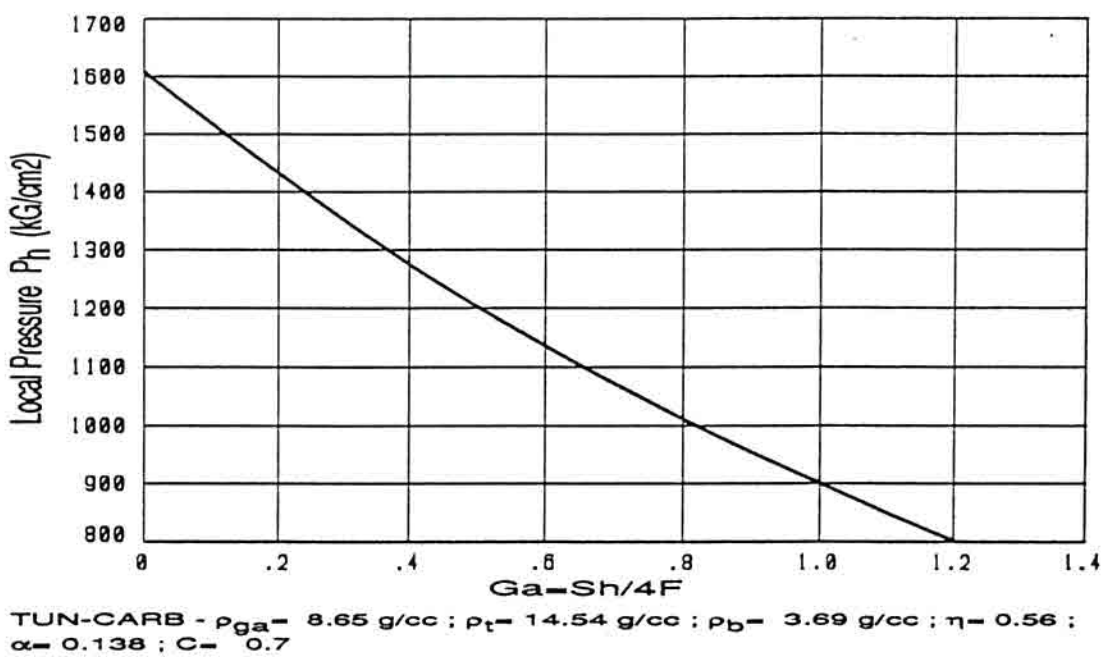
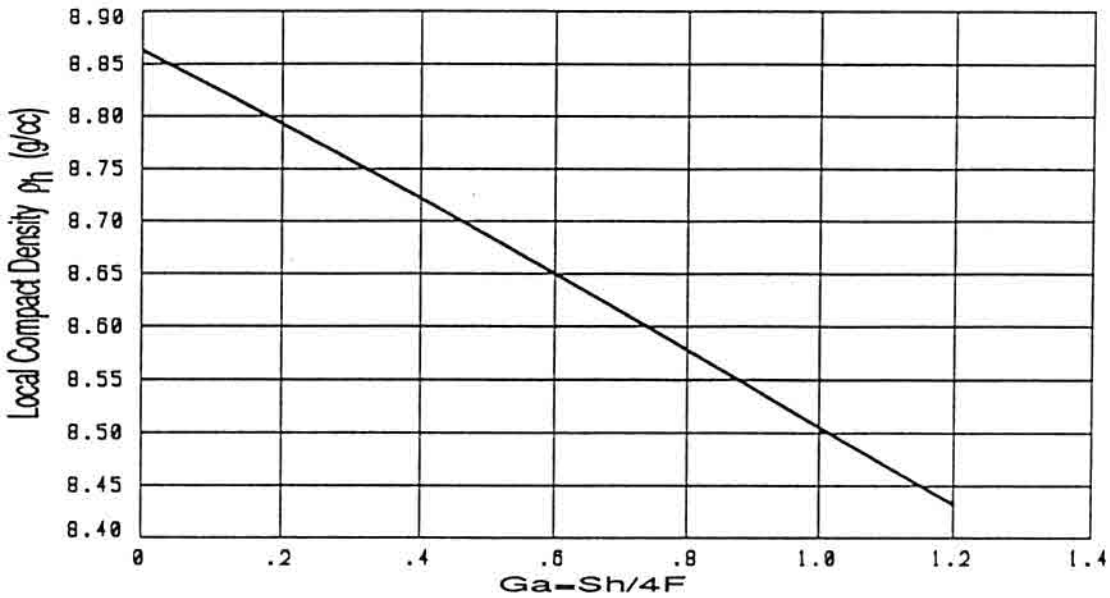
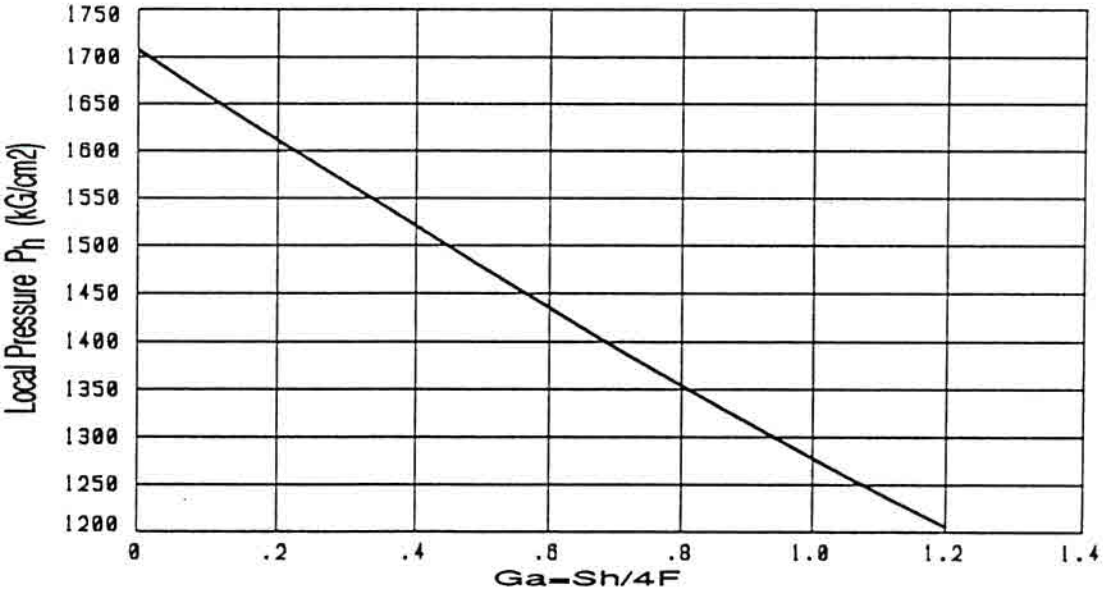


Figure 2. Local pressure distribution in a tungsten carbide compact during compaction.
ρga — average green density; ρ_t — theoretical density; ρ_b — bulk density



TUN-CARB - ρ_{ga}= 8.65 g/cc ; ρ_t= 14.54 g/cc ; ρ_b= 3.69 g/cc ; η= 0.56 ; α= 0.138 ; C= 0.7

Figure 3. Local density distribution in a tungsten carbide compact during compaction. ρ_{ga} — average green density; ρ_t — theoretical density; ρ_b — bulk density



BRON-IRO - ρ_{ga}= 6.40 g/cc ; ρ_t= 8.76 g/cc ; ρ_b= 3.15 g/cc ; η= 0.75 ; α= 0.163 ; C= 8.2

Figure 4. Local pressure distribution in a bronze-iron compact during compaction. ρ_{ga} — average green density; ρ_t — theoretical density; ρ_b — bulk density

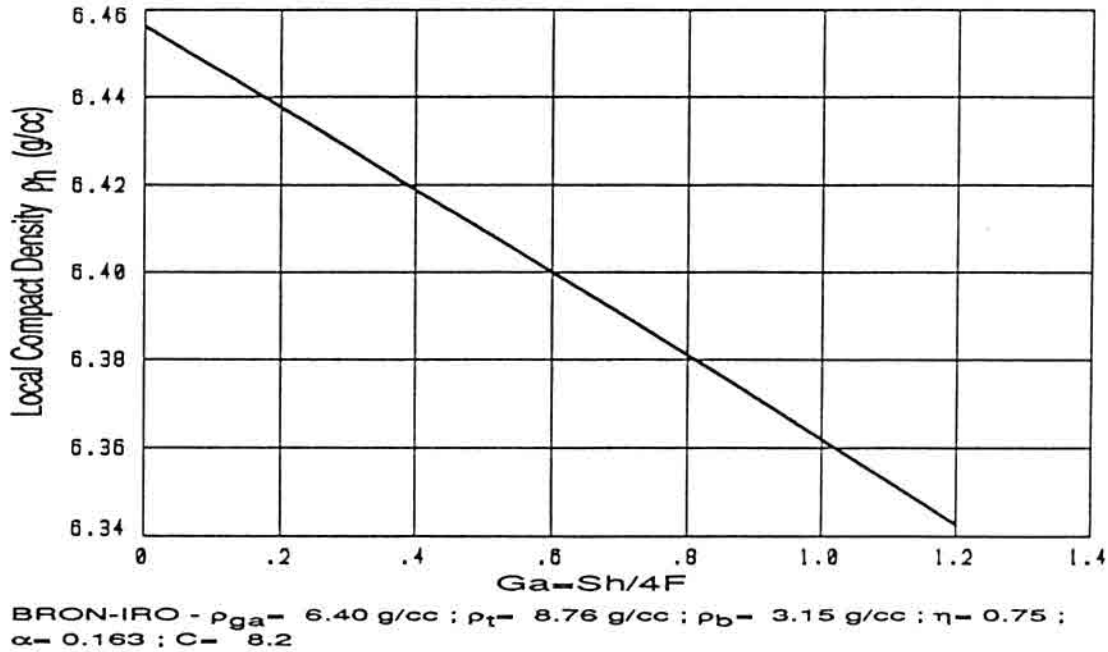


Figure 5. Local density distribution in a bronze-iron compact during compaction.
ρ_{ga} — average green density; ρ_t — theoretical density; ρ_b — bulk density

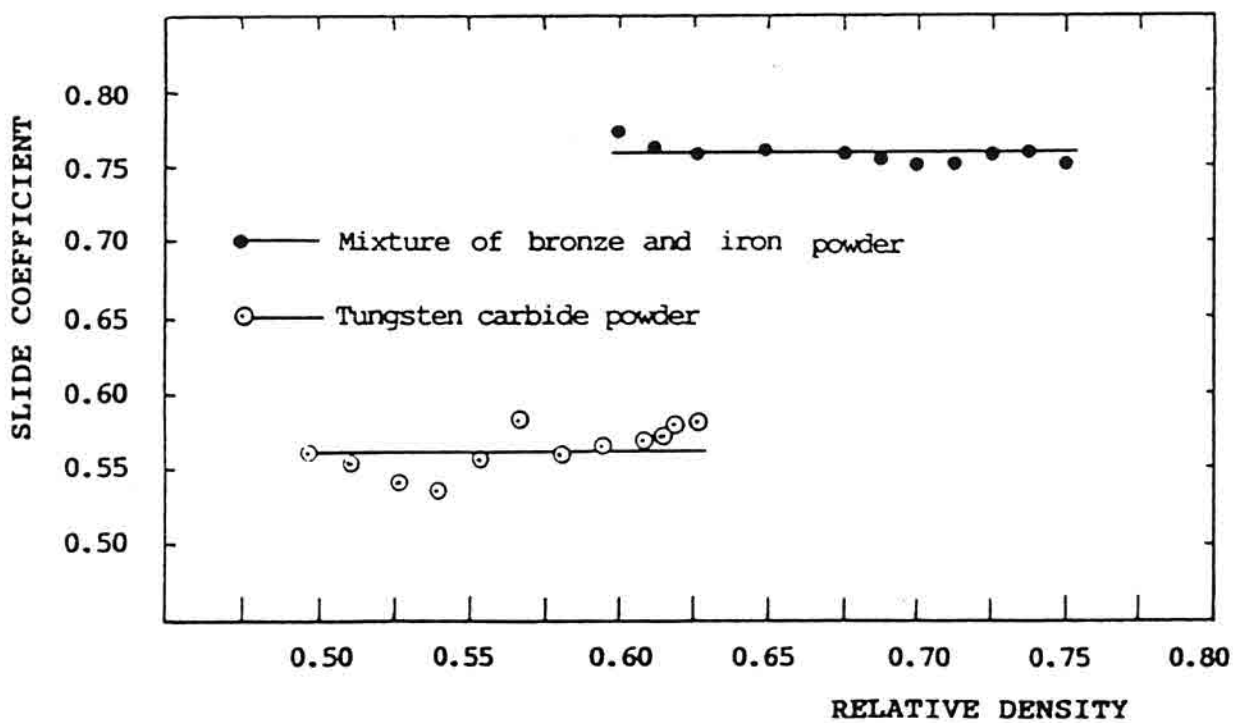


Figure 6. Slide coefficient as a function of a relative (green) density for tungsten carbide and bronze-iron compacts.

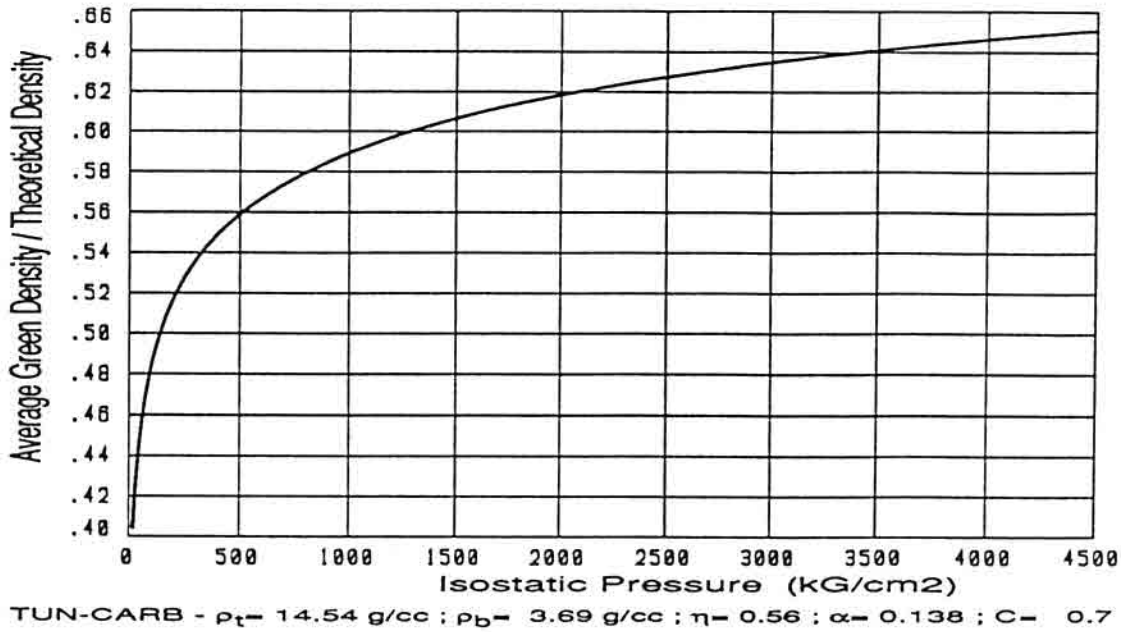


Figure 7. Relative (average) density as a function of a net pressure for tungsten carbide compacts.

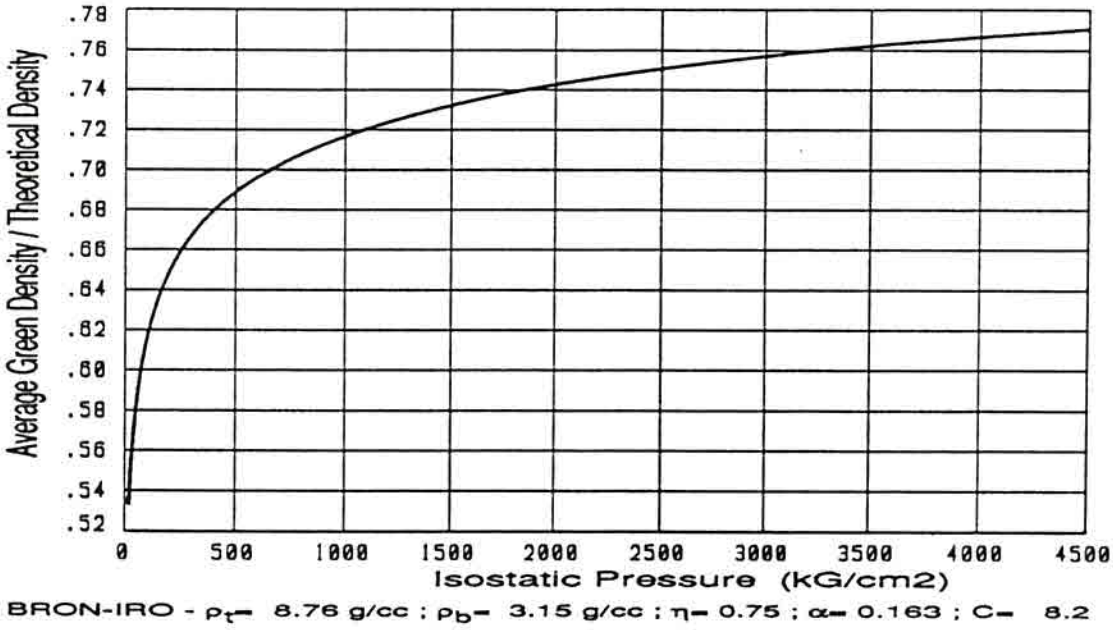


Figure 8. Relative (average) density as a function of a net pressure for bronze-iron compacts.

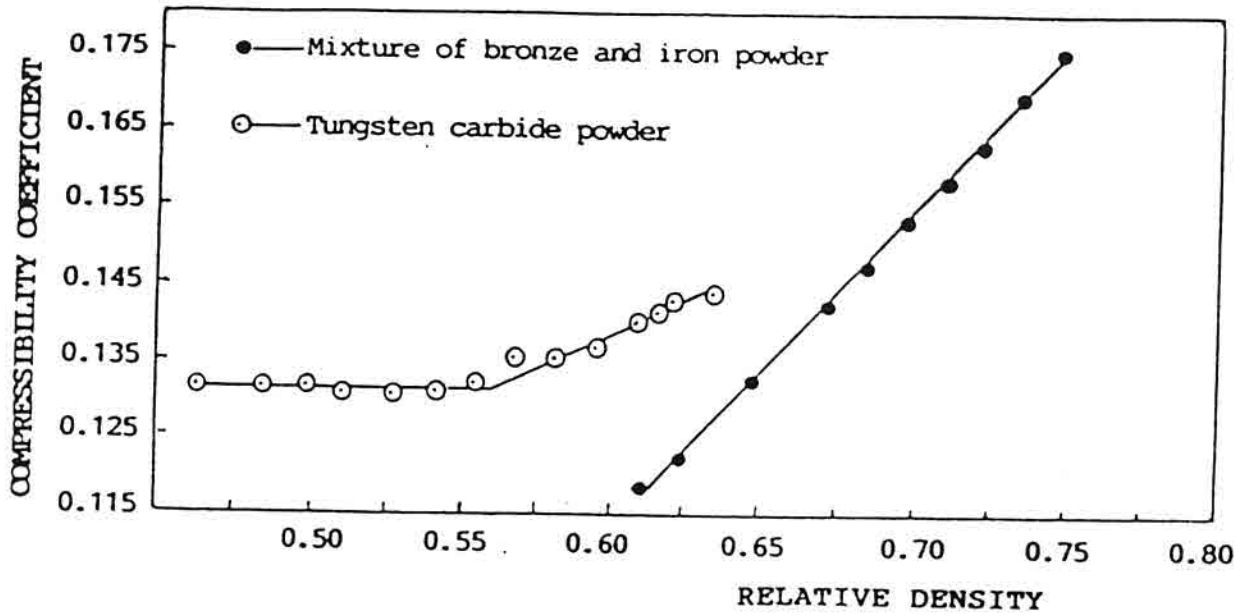


Figure 9. Compressibility coefficient as a function of a relative (green) density for tungsten carbide and bronze-iron compacts.

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COHESIVENESS OF A COMPACT

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Abstract

A procedure for determining the cohesiveness of a green compact obtained by powder pressing in a rigid die is discussed and confirmed by several experiments. An accurate estimation of the cohesiveness allows a reduction or elimination of cracks and laminations during production. It provides the tools for powder optimization during the design of a compaction process.

Introduction

The main objective of dry pressing is to produce from loose powders an agglomerate body with definite shape and strength allowing the preservation of the shape during ejection from a die, transportation and other technological operations prior to and during sintering. The set of parameters defining the mechanical strength of a compact is defined as the *cohesiveness* of an agglomerate [1].

The force interaction phenomenon observed between surfaces of the grains being in contact with each other was observed by Orr [2] and Morgan [3]. The work of Rumpf [4] established four principal mechanisms that govern the consolidation process of the grains. They are: *solid bridge formations, liquid bridge formations, binder aided consolidations, consolidations by Van der Waals or electrostatic forces.*

Powder grain consolidations by solid bridge formations occur even without any additions of binder and may be promoted by thermal phenomena similar to those encountered during sintering. Formation of solid bridges between grains can be initiated by crystallization or chemical processes. It should be emphasized that the above processes do not occur during cold die pressing.

The second type of bonding that may exist between the grains is the occurrence of liquid bridges developed in a presence of ceratin amount of low viscosity liquids. This type of bonding may occur in semi-dry pressing of ceramic powders. In a conventional die pressing of ceramic powders based on oxides, carbides, and nitrides, the bonds between the grains are most frequently formed through introduction of a liquid of high viscosity. Under pressure, the binder phase or any cementing agent is trapped in between the irregular surfaces of grains. The resulting strength of such an agglomerate is much higher than the strength of the binder phase itself [5].

The bonding of grains resulting from Van der Waals (non-metallic powders) or electrostatic forces (me-

tallic powders) are considered to be stable on condition that there is a large enough contact area between the grains.

On the other hand, real ceramic powders with rough surfaces and high deformation resistance will not form during pressing agglomerates of enhanced cohesion. The consolidation mechanism just mentioned can be applied to a powder system for which the grain size distribution is wide enough to alter the contact area between the particular grains.

At this point it should be emphasized that there exists yet another consolidation mechanism where the bonds between individual grains are formed by interlocking or anchoring process with intensity governed by the grain shapes. A brief analysis shows that it is necessary to introduce ceratin amount of a liquid to a ceramic powder system in order to get high consolidation level. Moreover, assuming that the mechanical interlocking has little influence on the compact strength (as demonstrated in various experiments), it would be difficult to consolidate a ceramic powder by die pressing without introduction of a binder phase. In contrast, most compacts pressed from metal powders attain high mechanical strength without binder addition.

The arguments presented above indicate a necessity to find a proper criterion for the estimation of the cohesiveness of a given compact. Such cohesiveness should be related to the ability of a powder to reach high strength after compaction. Many approaches aimed at establishing a general criterion to estimate the cohesiveness of a compact have been made without much success.

Cohesiveness and Die-Pressing Process

It is commonly observed that majority of defects such as cracks or laminations found in ferrite compacts before or after sintering are created during compact ejection from the die. An analysis of the ejection mechanism indicates that the mechanical parameters can serve as the best criterion for the evaluation of cohesiveness. The relationship between a compressive strength and compaction pressure for cylindrical samples is shown in Figure 1. The compacts were pressed from manganese-zinc ferrite powder containing different amounts of binder and lubricant.

Although the mechanical strength becomes higher as the compaction pressure increases, it appears impossible to press defect-free compacts from powders that contained the lowest amount of binder. It also has been observed that gradual elimination of the defects takes place when a friction is altered by addition of a zinc stearate known to serve as a good lubricant. It has been established experimentally [6] that the defects (cracks or laminations) appearing during compact ejection from the die can be avoided if the following inequality will be satisfied:

$$\frac{P_t}{F_b} < W_s \tag{1}$$

Here, P_t is the friction force equal to the ejection force, F_b is the lateral area of the compact and W_s is the compact green strength.

In general, the force balance that exists during compaction for a single action pressing can be written in the form:

$$P_c = P_d + P_t \tag{2}$$

where P_c is the force applied to the pressing punch, P_d is the force transmitted to the stationary punch and P_t is the friction force.

The relationship between the force applied to the pressing punch and the force transmitted to the stationary punch has been derived by Gasiorek and his co-workers [6,7,8] and takes the following form:

$$P_d = P_c \eta^{\frac{SH}{4F}} \tag{3}$$

where η is the sliding friction coefficient (slide coefficient) of the powder against the die wall, S is the perimeter of the cross-section of the compact, H is the height of the compact, and F is the cross-section area of the compact. *All dimensions are measured for a compact under full load inside the die.*

By combining equations (2) and (3), the friction force can be found as:

$$P_t = P_c \left(1 - \eta^{\frac{SH}{4F}}\right) \tag{4}$$

Inserting equation (4) into (1) leads to:

$$\frac{P_c \left(1 - \eta^{\frac{SH}{4F}}\right)}{F_b} < W_s \tag{5}$$

It is clear in the above expression that the right-hand-side of the inequality decreases when the slide coefficient approaches unity, i.e. $\eta \to 1$.

Moreover, the inequality (5) and the relationship shown in Figure 1 confirm the practical observation that an increase in the mechanical strength by raising the compaction pressure alone will not eliminate defects (cracks, lamination) appearing during compact ejection from a die. The defects in the compact can be eliminated through improvement of the powder ability to reach better consolidation during compacting.

The inequality (5) can be expressed in the following form:

$$\frac{W_s F_b}{P_c \left(1 - \eta^{\frac{SH}{4F}}\right)} > 1 \tag{6}$$

This indicates that the cohesiveness should be greater than 1 to prevent cracks in die pressed compacts.

Following the above analysis, the cohesiveness of die pressed compacts is determined as:

$$C = \frac{W_s F_b}{P_c \left(1 - \eta^{\frac{SH}{4F}}\right)} \tag{7}$$

The cohesiveness C is a nondimensional number and it should depend mostly on powder properties, material of a die, and die surface finish.

Experimental Procedures

Several experiments were conducted to study the cohesiveness of compacts pressed from the following powders: (a) tungsten carbide with 2% of wax (binder) and controlled amount of a zinc stearate serving as a lubricant; (b) steel (ANCOR 1000B) with controlled amounts of zinc stearate as a lubricant.

Several cylindrical compacts were made from each of these powders at *constant relative density* (average apparent density of a compact divided by the theoretical density of the powder) equal to 0.62 for tungsten carbide and 0.70 for steel. The addition of various amounts of lubricant changed the theoretical density and, accordingly, the average apparent density of the compacts. The compacts were pressed and then crushed in the computer controlled ***Powder Testing Center Model PTC-02DT*** (see acknowledgments). The slide coefficient and the green strength for each case were recorded. Two different die materials were used for tungsten carbide powders: steel die and tungsten carbide die.

Results and Discussions

Figure 2 presents the relation between cohesiveness and relative density of compacts pressed in a steel die from a tungsten carbide powder with 2% wax serving as a binder. The lower relative density range is limited by low green strength of compacts (on the order of 10 kG/cm^2), which must be sufficient enough to handle a compact without mechanical damage. The upper relative density range is limited by an excessive green expansion of compacts in the direction of pressing during removal from the die. Such excessive expansions can lead to laminations. The maximum green strength recorded for these tests reached magnitudes of 150 kG/cm^2.

Looking at the results in Figure 2 it is noticeable that the cohesiveness does not show clear dependence on the relative green density nor on the green strength (20-fold increase over the range). Based on a simple arithmetic averaging, the average cohesiveness for the results in Figure 2 is C_{avg}=0.655 with data spread of ±12.7 %. Therefore, for practical purposes the ***cohesiveness can be treated as a material constant independent from average green density of pressed compacts.*** As indicated earlier, all parameters related to green compacts are for compacts inside the die and under full load. This is essential to preserve a common ground for comparisons of various results [9,10,11].

As Figure 2 shows, the cohesiveness of tungsten carbide compacts with 2% of wax and pressed in a steel die is less than one. The green strength of such compacts is lower than friction forces inside the die. As a result, such compacts can develop cracks and laminations during ejection from a steel die unless special steps are implemented. Possible remedies include careful die designs and hold down pressure applied to compacts during ejection.

According to relations (6) and (7), the increase of cohesiveness can be achieved by increasing the slide coefficient (decreasing friction between powder and die walls). To demonstrate that dependence, several tests were performed with powders pressed in a die made of tungsten carbide. There, controlled amounts of lubricant were added to several compacts to change the slide coefficient. The results of those tests are demonstrated in Figure 3.

Comparing the results in Figures 2 and 3 it is clear that a change of die material from steel to tungsten carbide increased the cohesiveness approximately 2.5 times. In both cases the green strength of those compacts is on the same order; the parameter responsible for the increase in cohesiveness is the slide coefficient being larger by 0.16 for the die made of tungsten carbide than that made of steel. The addition of a lubricant further increases the cohesiveness due to both the increase in slide coefficient as well as the increase in green strength (Figure 4.). A close analysis of the results points out that the green strength of compacts increases with addition of a lubricant in amounts less that 0.2% by weight. Further addition of the lubricant does not seem to have positive effect on the green strength and can even cause its decrease.

Excessive use of lubricants for tungsten carbide powders can result in other negative effects such as a strong binding of green compacts to punches observed in tests with powders containing more than 0.2% of the lubricant. For powders with lubricants in amounts exceeding 0.5%, the bonding to punches was so strong that a separation of compacts from the punches resulted in severe mechanical damage to the compacts.

In case of steel powders, the behavior is somewhat different. The addition of zinc stearate as a lubricant results in a significant increase in cohesiveness (Figure 5). Concurrently, as demonstrated in Figure 6, an ad-

dition of zinc stearate results in an increase of slide coefficient (maximum tested addition was 0.5% by weight). At the same time, even small addition of a zinc stearate decreases significantly the green strength of compacts. This behavior is totally different from the one for tungsten carbide powders with binders.

Conclusions

The results presented in this study seem to confirm the validity and usefulness of the suggested way of defining cohesiveness for compacts obtained through powder pressing in rigid dies. The cohesiveness as defined here supplies valuable information on the ability of a given powder to form a strong aggregate under given compacting conditions (die material and die surface quality). The magnitude of the cohesiveness also depends on the side surface of the compact. It indicates that larger surface of a compact demands higher ability of a powder to form cohesive structures to prevent such compacting defects as cracks and laminations. For example, higher cohesiveness of metal powders allows manufacturing of thin wall cylinders or gears which in case of ceramic powders (oxides, carbides, nitrides, and the like) with binders would be very difficult or often impossible.

The proposed method of determining the cohesiveness of compacts pressed in rigid dies can be used to control powder preparation process, determination of optimum amounts of binders and lubricants as well as in research of new processes that will take into account optimum powder grain sizes, size distributions, shape distribution and others.

Acknowledgments

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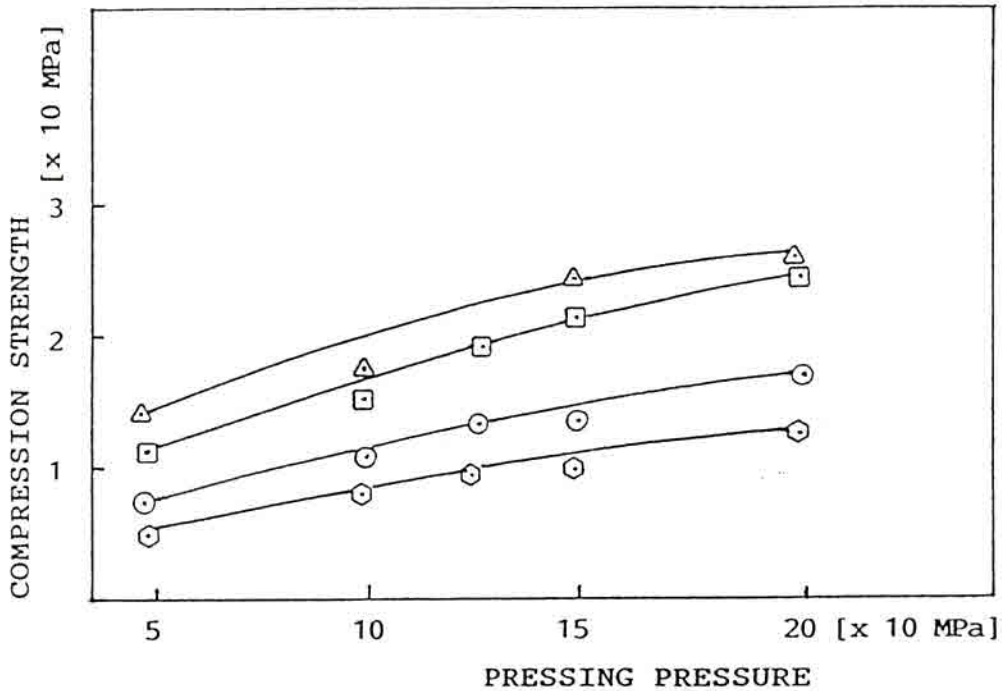

Figure 1. Green strength of a compact versus compacting pressure for Mn-Zn ferrite.
1 - 0.4 wt% PVA; 2 - 1.5 wt% PVA; 3 - 1.5 wt% PVA and 0.2 wt% zinc stearate; 4 - 2.0 wt% PVA

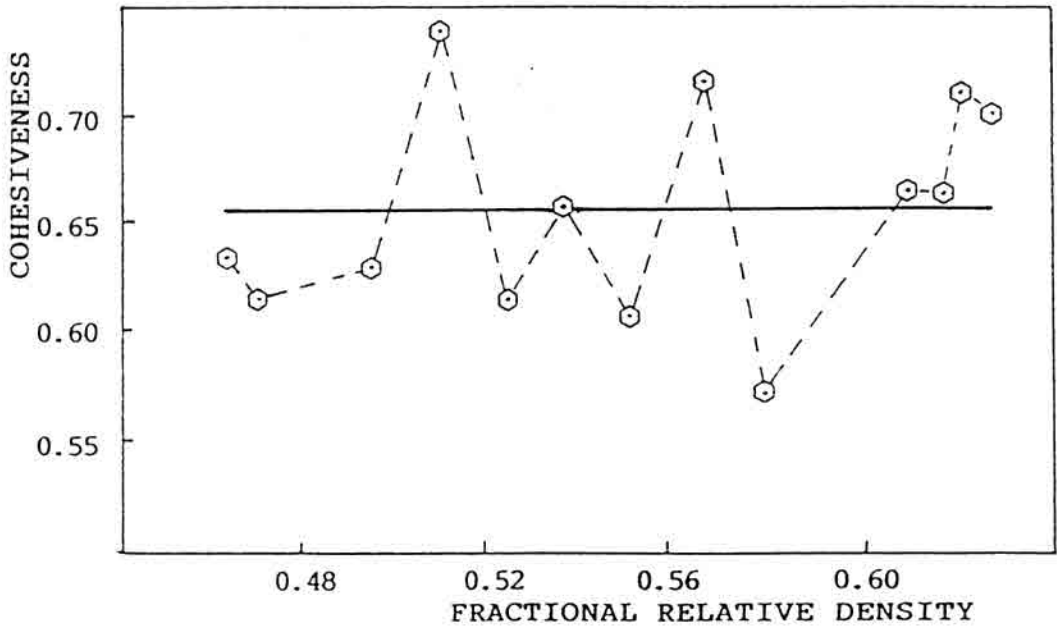

Figure 2. Cohesiveness versus relative density for tungsten carbide powders with 2% of wax pressed in a steel die.

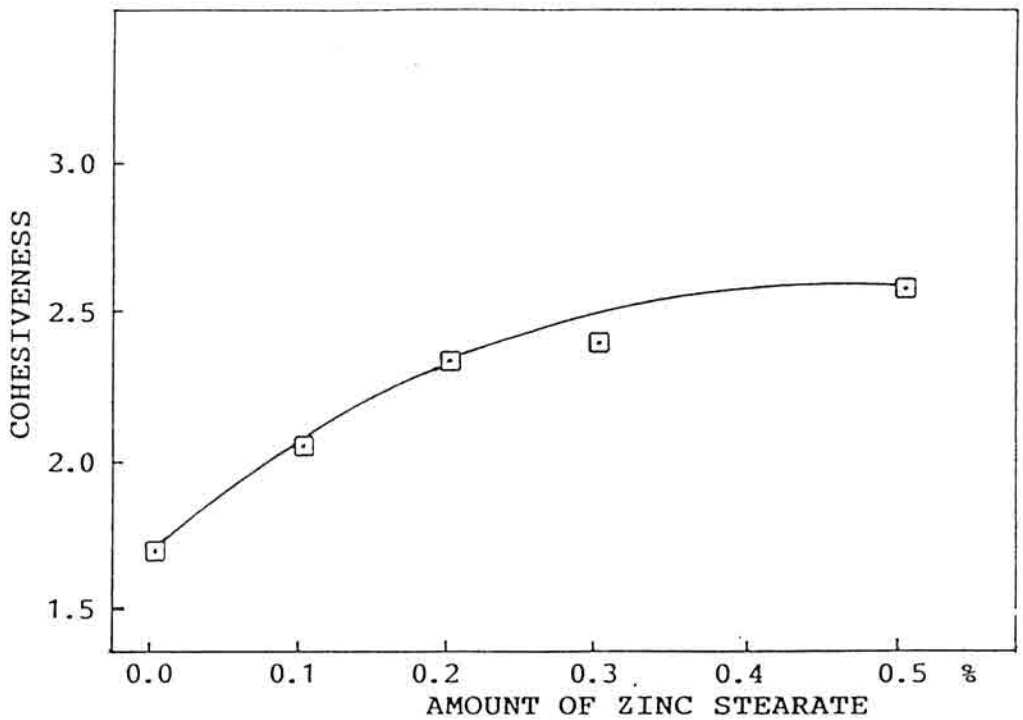

Figure 3. The effect of zinc stearate on cohesiveness of tungsten carbide powders pressed in a tungsten carbide die.

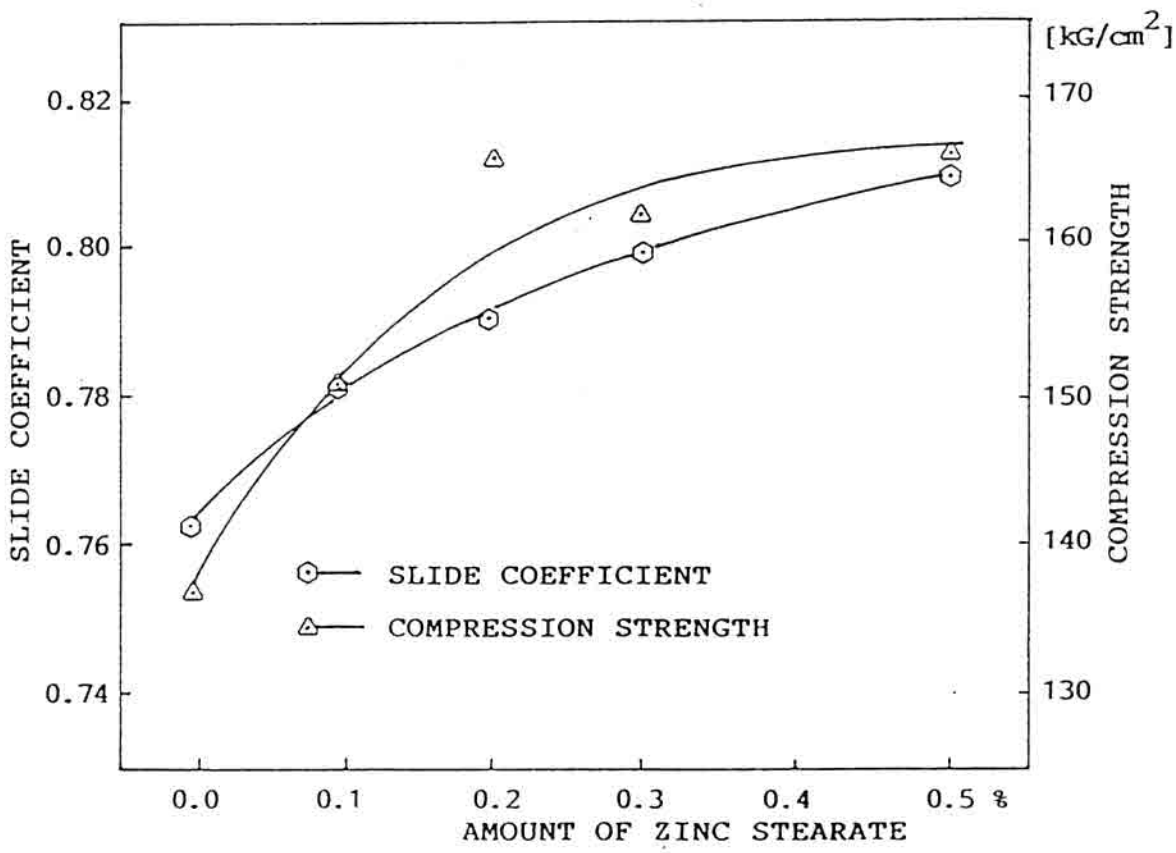

Figure 4. The influence of zinc stearate on slide coefficient and green strength of tungsten carbide compacts pressed in a tungsten carbide die.

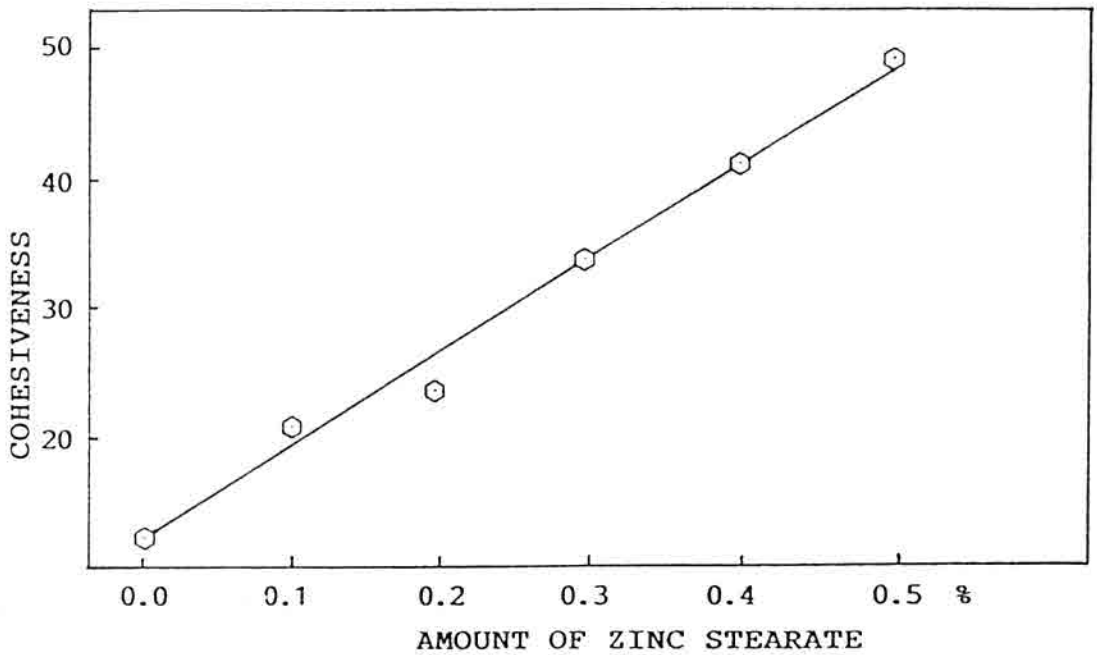

Figure 5. The influence of zinc stearate on cohesiveness for steel powders (ANCOR 1000B) pressed in a tungsten carbide die.

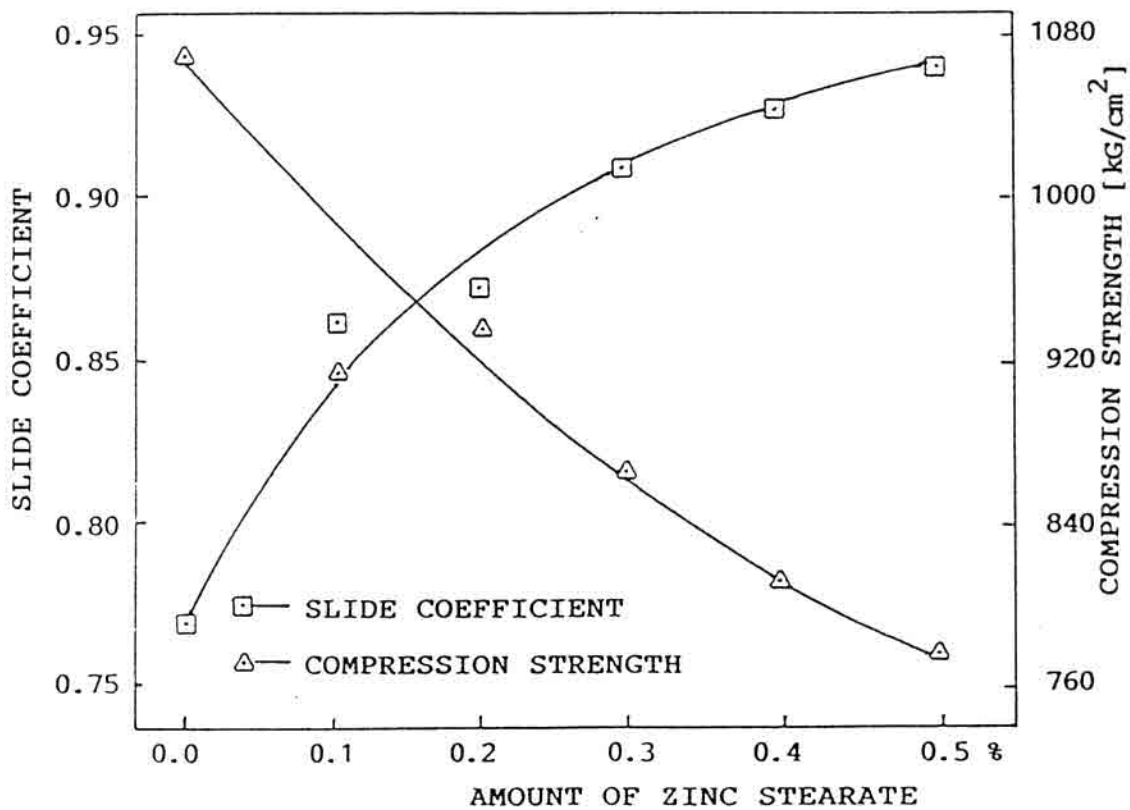

Figure 6. Slide coefficient and green strength of steel (ANCOR 1000B) compacts pressed in a tungsten carbide die.