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ADVANCED CEMENTED CARBIDES WITH ENHANCED FUNCTIONAL PROPERTIES FOR PM TOOLING APPLICATIONS.

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Functionally, the ability of pressing tools to retain component size within required tolerances and with a high surface finish allows the fabrication of intricate parts from alloyed powders.

During press compaction of powders and sizing operations of pre-sintered compacts, tools are subjected to intensive wear and corrosion from the working environment.

Therefore, the ability of the carbide tools to withstand various mechanical forces and corrosion plays a crucial role in the quality of press-shaping operations and directly affects manufacturing costs.

Recently developed cemented carbide grades demonstrate enhanced functional characteristics including lower ejection forces during compaction and exhibit superior corrosion resistance in their application as PM compacting and sizing tools.

A group of newly developed cemented carbide compositions for PM tooling applications are presented and their physical and functional characteristics are discussed.

Introduction.

The ability of powder metal (PM) technologies to provide end customers with the applications that demonstrate superior working characteristics combined with net-shape dimensions of the components is a distinct advantage, especially in large-scale manufacturing of cost-efficient and high quality products. In order to be successful in enhanced and productive output of PM parts production, stable cold compaction (pressing) operations are the primary processes which critically affect productivity and cost of the whole manufacturing cycle. For this reason, the compaction operation itself as well as its tooling segments, such as die sets and punches to be used on pressing equipment, get constant attention and are in the focus of operational specialists, engineering services and technologists at any PM facility worldwide.

During conventional press compaction of powdered compositions and sizing operations of pre-sintered compacts, tool members are often subjected simultaneously to intensive wear and aggressive corrosion attack from the working environment. Functionally, the ability of a pressing tool to retain its size with required tolerances and high surface finish allows the most productive and cost efficient method of fabricating intricate parts from alloyed powders. At the same time, serviceability of the compaction tooling to a large degree depends on how susceptible those tools are to corrosive media during fabrication, e.g. shaping by EDM processing, etc. Understandably, it is also important for PM tooling to resist the aggressive working environment, when dies, core rods, pins and punches are interacting with sulphur or chlorine radicals, etc, from the solid lubricants or additives that almost every powdered blend contains within it. Therefore, the ability of the tool elements to withstand various mechanical forces and corrosion attacks while providing lower ejection forces plays a crucial role in the quality of press-shaping operations and directly affects manufacturing costs. Traditionally used materials, such as alloyed steels and tool steels [1], Ferro-TiC hard alloys (high TiC-containing steel- or other alloy-bonded composites) and cemented tungsten carbide-cobalt (WC-Co) hardmetal grades [2] are not always able to fulfil the challenges present during the compaction of metal powders and its production environment.

In this work, a group of cemented carbide grades recently designed and developed by General Carbide Corporation (Greensburg, Pennsylvania. USA) for PM compaction applications has been tested and investigated on a comparative basis with respect to their friction characteristics during cold compaction of various powdered compositions. This tribological study took place at the R&D Center of GKN Sinter Metals, located in Radevormwald / Germany. Other functional as well as physical and mechanical properties of the carbide grades were also explored in the search for a more intelligent approach towards selection of efficient materials for PM compaction and /or sizing tooling applications.

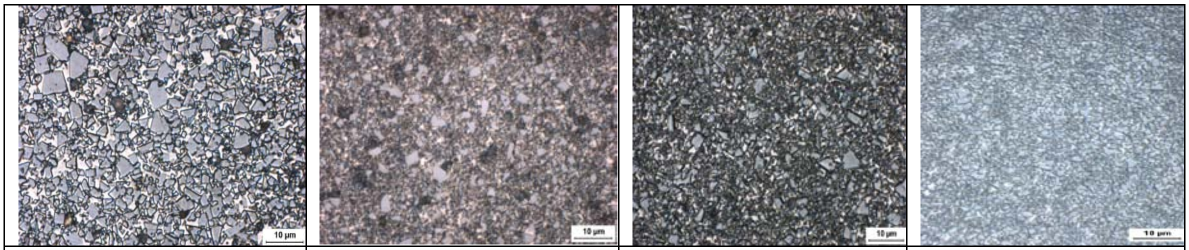
Utilized Materials and Alloying Concept Applied in Carbide Grades for PM Tooling Applications.

For PM compaction tool applications, several medium-binder grades recently developed at General Carbide Corporation for PM tooling applications, were selected. Those carbide grades, namely, GC-813CT and GC-613CT having approximately the same amount of cobalt binder (Table.1) use a similar alloying concept utilizing tantalum carbide (TaC) and chromium-based additives. The work environment, combines atmospheric corrosion and high mechanical stresses with intensive adhesive scuffing and abrasive wear. Two additional grades were chosen for comparative purposes. GT30 from Kennametal Widia is frequently used in compaction die operations [2], and GC-313 is a product of General Carbide Corporation [3]. The latter has no alloying additives and is a straight WC-Co composition. The properties of both GC-313 and GT30 are also presented in Table 1.

Table 1. Chemical Content and General Mechanical Characteristics of Selected Carbide Grades.

Grade ID	Chemical Content, w/%	Average Grain Size (micron)	Density, g/cc	Hardness, HRa	Transverse Rupture Strength, MPa	Comments
GC-813CT	WC-bal. TaC-2.0 Cr_3C_2-1.00 Co-13.00	1.0 & 4.5 mixture.	14.24-14.36	90.5-91.5	3172	Consolidated by Sinter-HIP process
GC-613CT	WC-bal. TaC-3.00 Cr_3C_2-0.75 Co-13.00	6.0	14.01-14.13	87.4-88.4	3206	Consolidated by Sinter-HIP process
GC-313	WC-bal. Co-13.00	3.0	14.15-14.27	88.1-89.1	3550	Consolidated by Sinter-HIP process
GT30	WC-bal. TiC-1.60; Co-15.00	2.0	14.00	87.8-89.2	2703	

The microstructures of the four carbide grades are shown in Figure 1.



GT-613CT	GC-813CT	GT-313	GT30 (KM Widia)

Figure 1. Microstructure of Selected Carbide Grades (1550x).

The rational for alloying WC-Co cemented carbide with tantalum- and chromium-containing additives was based on results in the literature [4-7]. Additions TaC to WC-Co cemented carbides by Ettmayer [4] provided beneficial effects for both cutting and structural tool applications, attributed to higher hot hardness. Samsonov et. al. [5] confirmed that at 730°C the hardness of TaC was almost 2 times higher than WC, even though the hardness of WC at room temperature is 50% higher than that of TaC (2400 kgf/mm^2 vs.1570 kgf/mm^2, respectively),.

Moreover, it was found that small additives of TaC lead to an increase in the strength of hardmetals as well as enhancing the toughness of the carbide-binder [6.7]. Also, a small addition of TaC to WC-Co alloys inhibits the recrystalization grain growth of the carbide phase during the sintering process, and therefore the resulting alloys demonstrate a tendency to have finer and more uniform grains

However, the most important reason for using TaC additives is its pronounced resistance to galling wear. When specific tribological conditions occur between metallic surfaces, the softer member within the friction couple tends to adhere to the surface of the harder metal due to the large contact stresses and increased temperatures at asperity contacts. Adherence of the work material to the die or punch may be also caused by excessive heat build-up being associated with friction between tool members or between tools and compacted powder within the die set. It was found that 1-2% TaC addition to WC-Co improves the resistance to micro-welding [3, 6, 8]. Adding Cr_2O_3 as a grain growth inhibitor at the co-carburization stage of WC processing as well as the addition of Cr_3C_2 into the cemented carbide formulation results in improved corrosion characteristics. Some authors [3, 6-8] attribute this to the formation of a protective complex oxide (especially, together with additives like TaC), and prevents solution of WC into the Co binder.

General Characterization and Testing Methods.

General characterization and testing of the selected materials was performed.

The microstructure was analyzed using optical microscopy at a magnification of x1500 after etching in Murakami's reagent. Rockwell hardness testing was performed according to ASTM B 294-92 while transverse rupture strength (TRS) was measured in compliance with ASTM B406-96. Density measurements followed the recommendations of ASTM B311-93.

Palmqvist toughness testing of selected carbide compositions was performed using a Zwick 3212 indenter. The Vickers indentation was made at loads ranging from 250-350 N (25-35 Kg*force) range. The lowest force that produced defined indents and cracks was used. Vickers hardness (HV) was calculated in accordance with ASTM E92 and E384 and Palmqvist fracture toughness (WK) was calculated by using the formula recommended in the literature [9-11], and ISO 28079:2009 (E) [12],

In addition to Palmqvist results, supplementary tests were performed to obtain the elastic modulus (E). Ultrasonic testing was performed on TRS bars to obtain longitudinal and shear velocity wave measurements via transducers. A Tektronix oscilloscope was used to measure the echoes and number of reflections. From these data and the material densities, the shear modulus G and P-wave modulus could be calculated. Then, these parameters entered into Lamés (**λ**) first parameter equation, which was then calculated and input into a final elastic modulus equation [Eq.1] for determining elastic modulus according to recommendations from several research works [13-14], where **λ** is Lamés first parameter and **G** is the Shear Modulus.

$$E = \frac{(3 \times \lambda) + (2 \times G)}{\lambda + G} \qquad \{1\},$$

Complementary, the coefficients of thermal expansion (CTE) were measured using an Anter Unitherm pushrod dilatometer model 1161V in accordance with ASTM E831. A thermal ramp rate of 5°C per minute to 800°C was used in a flowing ultra high purity argon atmosphere.

Results and Discussion

The measured data are presented in the Table 2. GC-813CT has the highest hardness while GC-613CT has the lowest hardness. Many authors [3-8] link high hardness to high wear resistance, especially to abrasive and sliding wear of carbide materials. Also, the CTE of the GC-813CT and GC-613CT grades had the lowest values. The CTE can be associated with a tool's ability to retain dimensional stability, when intensive friction forces may sizably raise the temperature on tool surfaces during a powder compaction process. The highest fracture toughness value was obtained for GC-613CT following by GC-313, whereas GT-30 and GC-813CT demonstrated a measurably lower level of toughness. Various independent studies [9-

10] show that the toughness (the ability of a material to resist crack propagation) affects fatigue properties of materials which is important for PM tooling applications, where multi-stroke cycling of tools is sustained through long-term use.

Other mechanical properties shown in Table 3, inconclusive in differentiating between the grades, but from a combination of TRS and modulus of elasticity, GC-313 provides the best result.

Table 2 . Tested Characterization Data for Selected Carbide Grades.

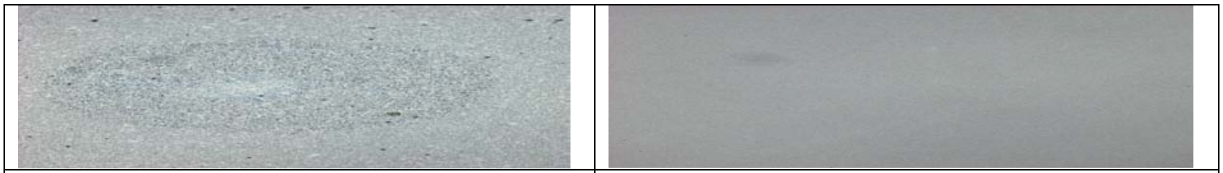
Grade's ID	Rockwell Hardness (Scale A), HRA	HV *(kgf/mm^2),* HV30	Palmqvist Fracture Toughness, W_K MN * (m^(-3/2))	Average CTE, 10^{-6} °C @ [RT-800 °C]
GC-813CT	**90.5 -91.5**	**1420-1505**	**13**	**5.87**
GC-313	**88.1-89.1**	**1180-1280**	**18**	**6.26**
GC-613CT	**87.4-88.4**	**1110-1210**	**23**	**6.15**
GT30	**87.8-88.8**	**1150-1250**	**14**	**6.50**

Table 3 Mechanical Properties Data for Selected Carbide Grades

Grade's ID	Average Mechanical Properties.			
	Average Transverse Rupture Strength, MPa	Mean Ultimate Tensile Strength*, MPa	Ultimate Compressive Strength, MPa	Modulus of Elasticity (E), GPa
GC-613CT	3206	2196	4405	**551**
GC-313	3551	2432	4234	**551**
GC-813CT	3172	2172	4550	**558**
GT30	2700	1850	4100	**540**

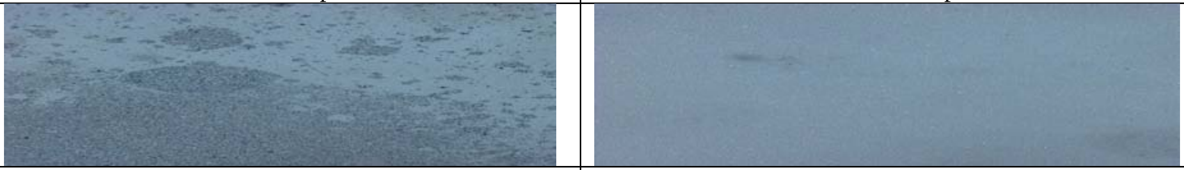
* ***Mean Ultimate Tensile Strength has been calculated upon General Carbide "The Designer's Guide To Tungsten Carbide" manual [see Website: www.generalcarbide.com]***

However, mechanical properties alone cannot be considered as the only criteria for selection of the best material for PM moulding tool applications. As an illustration of this, Figure 2 shows images of the sample surfaces after a comparative corrosion test, which was performed for GC-313 and GC-813CT grades.



GC-313: Test conducted in tap water over 48 hours.

GC-813CT: Test conducted in tap water over 48 hours.



GC-313: Test conducted in EDM-wire tank for 100 hours.

GC-813CT: Test conducted in EDM-wire tank for 100 hours.

Figure 2. Images of the Sample Surfaces after Comparative Corrosion Runs.

One group of samples was corroded in low-chlorinated tap water, whereas the other group of samples was put into EDM-wire tank liquid containing various deflocculants and chlorine radicals for 100 hours. Figure 2 demonstrates the improved corrosion resistance of GC-813C with (Ta, Cr) C additives versus the GC-313 grade.

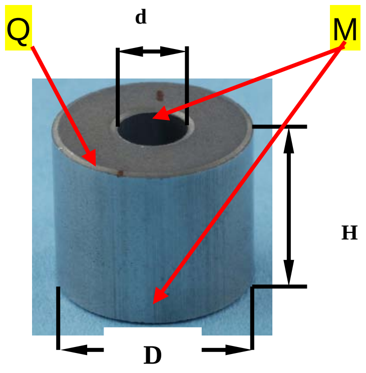
Tribology Study of PM Die Inserts from Selected Carbide Grades

Specific testing to reproduce real moulding conditions in cold compaction of metallic powders was performed in order to evaluate the behaviour of die inserts made from the grades under evaluation.

a) Experimental Procedure.

Frictional characteristics were tested and evaluated on specialized computerized press equipment set at the R&D Center of GKN-Europe (Radevormwald, Germany). The grades were fabricated into carbide inserts, and afterwards assembled into compaction dies and installed into the experimental press for subsequent tribological evaluation of the friction forces. The parameters are given in Figure 3. Cylindrical bushings of lubricated iron-based powder were compacted in die sets equipped with the carbide inserts (Figure 3a). The green compacts had the following dimensions: external diameter (D) of14 mm; internal diameter (d) of 9 mm; and height (H) 12 mm. These dimensions correspond to M/Q ≥10 based on ca. 2 (Figure 3b) in accordance with the recommendation of work [15]. As seen from Figure 3c, various M/Q ratios strongly affect the friction properties of the tools.

The distribution of friction forces during compaction was measured. Tests were performed at 22°C with 800 MPa applied pressure on the upper punch. Presented data are mean values from 5 measurements.

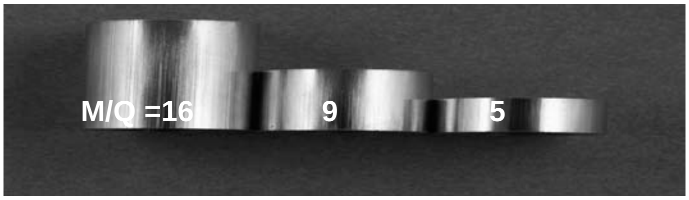


a) Bushing-shape green compact from iron-based powder blend.

b) Tribology Test Parameterization Conditions.

For compaction, the ratio of sliding surfaces M to pressing face Q is critical for the friction and pressure distribution.

$$\frac{M}{Q}=\frac{\sum M_i}{Q}=\frac{\pi(D+d)H}{\frac{\pi}{4}(D^2-d^2)}=\frac{4H}{D-d}\geq 10 \qquad \{2\}$$



c) Iron green compacts at various M/Q ratios.

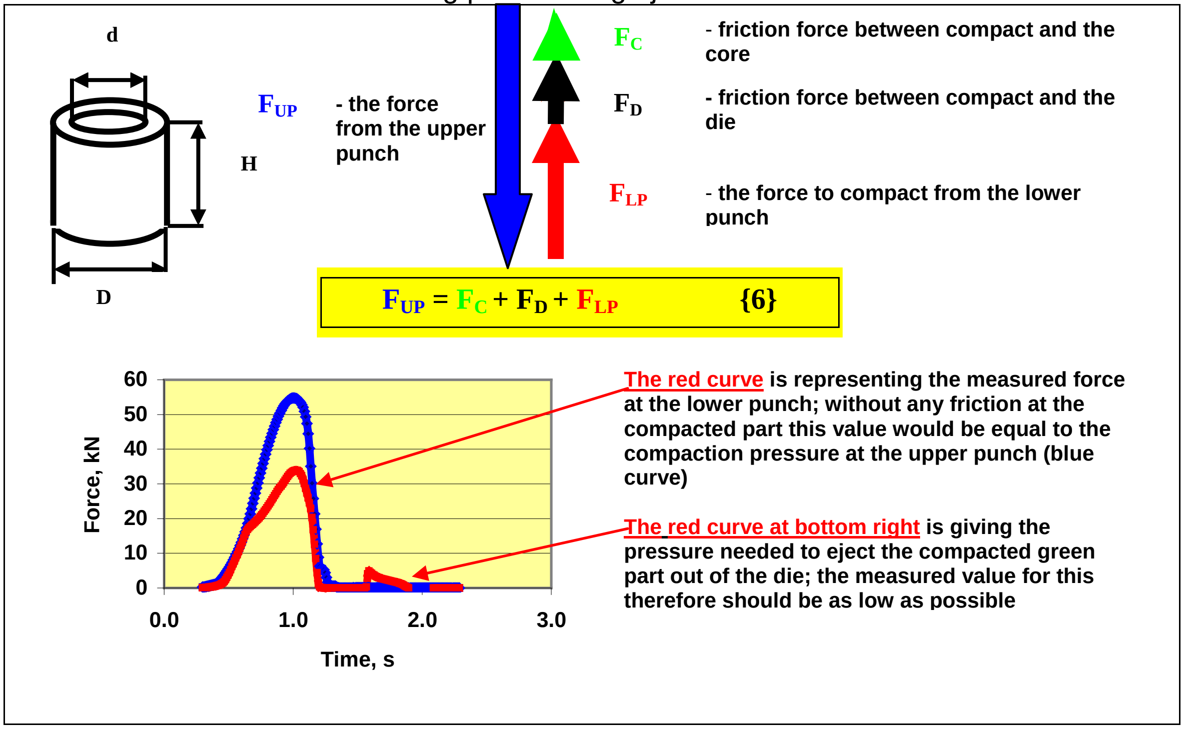
Figure 3. Shape of Iron-Based Green Compact and Tribology Test Parameterization Conditions.

The equipment allowed the compaction pressure at the upper punch and the bottom punch as well the ejection force during compaction to be recorded. This is schematically shown in Fig, 4 as well as a summary of the forces measured during testing in a single compaction stroke

The red curve represents the measured force at the lower punch. Without any friction at the compacting part, this value would be equal to the compaction pressure at the upper punch (blue curve), i.e. at such “ideal” frictionless scenario **F_{UP} = F_{LP}**. However, in reality, the disparity between the forces applied to various tool members always exists due to the friction between the tooling and compacted part.

The difference between the **F_{UP}** and **F_{LP}** values means that the higher **F_{LP}** value (i.e. the smaller the variance between forces applied to the upper and bottom punches), the better the friction properties of the tool. This is because less non-productive force loss occurs in the die (**F_D**) or other tool members (**F_C**) The peak on the red curve at bottom right in Figure 4 is the pressure needed to eject the compacted part out of the die.

It is important to emphasize that the lower the pressure exhibited by the lower punch at the ejection stage, the better is the efficiency of the compaction process itself, derived from the fact that less frictional losses are taking place during ejection.



The red curve is representing the measured force at the lower punch; without any friction at the compacted part this value would be equal to the compaction pressure at the upper punch (blue curve)

The red curve at bottom right is giving the pressure needed to eject the compacted green part out of the die; the measured value for this therefore should be as low as possible

Figure 4. Schematic of the Friction Forces being Involved in Bushing Compaction Process

Also, as it is known from compaction practice [15-17] that the lower the ejection pressure is, the more suitable it is to perform compaction of fragile parts of complicated shapes or of powder materials with low green strength

b) Comparative Cold Compaction Test

Initially, two dies with GT30 and GC-813CT were subjected to a comparative compaction trial under identical testing conditions using double-sided compaction. The friction forces (including force applied on both top and bottom punches as well as ejection force during compaction) were measured and recorded. An iron-based blend containing A 1000B iron powder with 2% of copper and 0.8% of LicowaxC lube was compacted. The batch of thousand pieces of the bushing-type components was pressed in each case.

Figure 5 shows that the same level of force was observed in the compaction stroke with both the GT30 and GC-813CT carbide inserts.

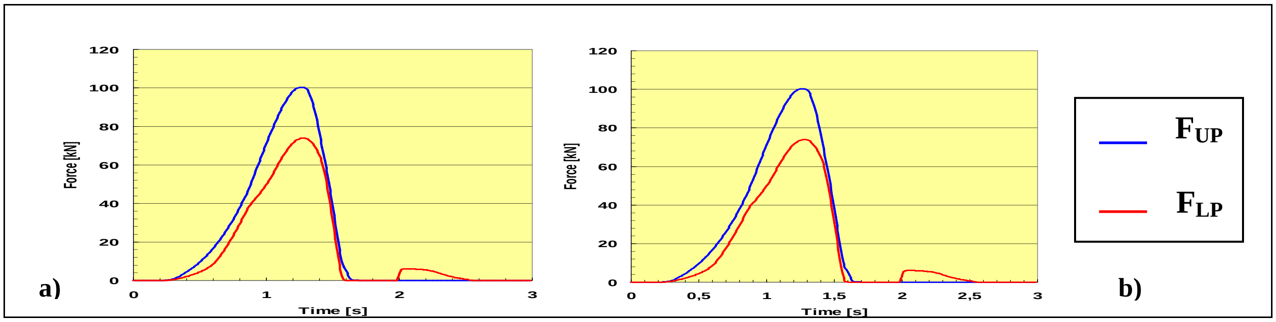


Figure 5. Force Records at Top (F_{UP}) and Bottom (F_{LP}) Punches for GC-813CT (a) and GT30 (b) Die Sets.

However, the difference between the two carbide inserts was observed in the measured ejection force, as seen in Figure 6.

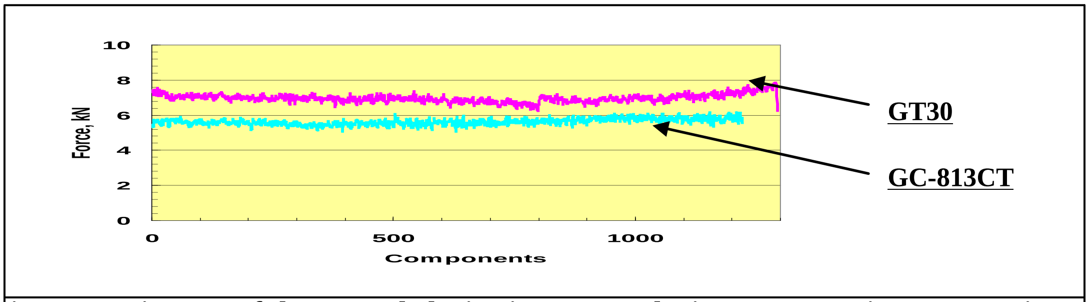


Figure 6. Diagram of the Recorded Ejection Forces during Comparative Compaction Run of Dies

Thus, a 15% lower ejection force (better ejection properties) was measured with GC-813CT insert in comparison to alike insert from GT-30 grade.

In the second series of tests, grades GC-813CT, GC-613CT, and GC-313 were compared to GT30. Compaction dies with carbide inserts from these materials were sent again to the R&D Center of GKN-Europe for comparative runs under similar testing conditions. In this case the press had a more rigid set-up with a single sided compaction mode (in comparison to dual punch compaction mode applied in the previous test) acting at a slower punch speed. The press was equipped with load cells at both the upper and lower punch, and at the die support. Two iron-based powder mixtures with lubricants and additives were chosen that in production exhibited frequent problems associated with compaction. The first blend, an iron- phosphorus material, is well known for its abrasiveness [16], while the second blend, namely, PM stainless austenitic steel, demonstrates both abrasiveness and a tendency to adhere to moulding tools [17].

The compositions of those blends are given below:

Blend #1: {Fe + 0,45% P + 0,25% MnS + 0,8% Acrawax} material;

Blend #2: {SS 316L + 0,8% Acrawax}.

Tables 4 and 5 contain the recorded values for lower punch pressure ($\mathbf{P_{LP}}$), and ejection pressure ($\mathbf{P_{eject}}$) respectively.

The highest $\mathbf{P_{LP}}$ value (indicative of lower frictional losses within the compaction tool assembly) was found to be for the die set equipped with GC-613, being closely followed by grade GC-813CT. The lowest $\mathbf{P_{LP}}$ values were recorded for inserts from GC-313 and GT30.

Table 4. Lower Punch Pressure (P_{LP}) Data Recorded in PM Die Set Equipped with Selected Carbide Inserts.

Grade ID	Lower Punch Pressure, P_{LP} (MPa)				Normalized Range,%
	Carbide Die Insert Grade				
	GC-313	GC-613CT	GC-813CT	GT30	
Blend #1	**575**	**595**	**590**	**590**	**6**
Blend #2	**366**	**378**	**375**	**370**	**10**

Referencing data shown in the Table 5, GC-613CT grade demonstrates the lowest ejection forces, $\mathbf{P_{eject}}$ for both compacted blends, being closely followed by grades GC-813CT and GT30, whereas the highest ejection pressure was measured with the GC-313 die insert.

Table 5. Ejection Pressure (P_{eject}) Data Recorded in PM Die Set Equipped with Selected Carbide Inserts.

Grade ID	Ejection Pressure, P_{eject} (MPa)				Normalized Range,%
	Carbide Die Insert Grade				
	GC-313	GC-613CT	GC-813CT	GT30	
Blend #1	**119**	**110**	**115**	**112**	**24**
Blend #2	**389**	**379**	**382**	**384**	**7**

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Summary

1) Tantalum carbide and chromium-based additives in the recently developed GC-813CT and GC-613CT carbide grades has positively affected mechanical properties and resulted in

superior performance in a corrosive environment versus straight WC-Co grades such as GC-313.

2) PM compaction dies equipped with inserts of various cemented carbide materials were tested during a special tribological study at GKN-Europe Technology Center (Radevormwald /Germany). Compaction testing found that better tribological characteristics were measured for PM molding tool sets equipped with die inserts made from GC-613CT and GC-813CT grades. This included lower ejection forces and smaller frictional losses.

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