Engineering
Materials

# SPECIALTY CARBONS FOR POWDER METALLURGY AND HARD METALS

TIMREX®
TIMCAL Graphite

ENSACO®
TIMCAL Carbon Black

imerys-graphite-and-carbon.com

IMERYS
Graphite & Carbon

# Imerys Graphite & Carbon

## WHO ARE WE?

IMERYS Graphite & Carbon has a strong tradition and history in carbon manufacturing. Its first manufacturing operation was founded in 1908.
Today, IMERYS Graphite & Carbon facilities produce and market a large variety of synthetic and natural graphite powders, conductive carbon blacks and water-based dispersions of consistent high quality.
Adhering to a philosophy of Total Quality Management and continuous process improvement, all Imerys Graphite & Carbon manufacturing plants comply with ISO 9001:2008.
IMERYS Graphite & Carbon is committed to produce highly specialized graphite and carbon materials for today's and tomorrow's customers needs.
IMERYS Graphite & Carbon belongs to IMERYS, the world leader in mineral-based specialties for industry.

## WHERE ARE WE LOCATED?

With headquarters located in Switzerland, IMERYS Graphite & Carbon has an international presence with production facilities and commercial offices located in key markets around the globe. The Group's industrial and commercial activities are managed by an experienced multinational team of more than 430 employees from many countries on three continents.

For the updated list of commercial offices and distributors please visit www.imerys-graphite-and-carbon.com

**Lac-des-Îles, Canada**
Mining, purification and sieving of natural graphite flakes

**HQ Bodio, Switzerland**
Graphitization and processing of synthetic graphite, manufacturing of water-based dispersions, processing of natural graphite and coke, and manufacturing and processing of silicon carbide

**Changzhou, China**
Manufacturing of descaling agents and processing of natural graphite

**Terrebonne, Canada**
Exfoliation of natural graphite, processing of natural and synthetic graphite

**Willebroek, Belgium**
Manufacturing and processing of conductive carbon black

**Fuji, Japan**
Manufacturing of water-based dispersions

## WHAT IS OUR MISSION?

To promote our economic, social and cultural advancement with enthusiasm, efficiency and dynamism by offering value, reliability and quality to ensure the lasting success of our customers.

## WHAT IS OUR VISION?

To be the worldwide leader and to be recognized as the reference for innovative capability in the field of carbon powder-based solutions.

# Our value proposition

We at IMERYS Graphite & Carbon deliver tailormade solutions for PM and Hard Metals applications with superior consistency of key products' parameters: Purity, Crystallinity, Particle Size Distribution, Oversize Control.

## TIMREX GRAPHITE POWDERS FOR POWDER METALLURGY

Graphite powders are extensively used in PM mixes, for two main technical purposes:

| PURPOSE OF GRAPHITE | PM MATERIALS | APPLICATION FIELD EXAMPLES |
|---|---|---|
| **Hardening by diffusion into Fe-matrix** | **Fe-based PM grades** | **Structural, engineering components** |
| Solid state lubrication and friction moderation | Cu/bronze-PM grades | Self lubricating engineering parts: bearings, bushes, valve guides, valve seats |
| | Fe-based PM grades | Friction materials: sintered brake pads, clutch facings, linings |
| | High alloy steels | Cutting tools |

It is possible to summarize the key requirements of PM Parts Manufacturing in four interconnected targets that must be addressed by this Industry - the 4 P's of Powder Metallurgy:

| KEY REQUIREMENTS OF PM PARTS MANUFACTURING | TECHNICAL REQUIREMENTS INVOLVED | BENEFITS FROM IMERYS GRAPHITES |
|---|---|---|
| **PRECISION**<br>Tight dimensional control (in-lot and lot-to-lot)<br><br>**PERFORMANCE**<br>High mechanical strength<br><br>**PRODUCTIVITY**<br>High parts/minute rate, minimized scrap/out of spec rate<br><br>**PRICE**<br>PM parts' cost competitiveness versus other materials and manufacturing technologies | • Good mixability, low tendency to segregation<br>• Dust-free handling<br>• Good flowability, in terms of high flow rate and flow consistency<br>• Low wear of compaction tools & dies<br>• Low and consistent dimensional change during sintering (in-lot and lot-to-lot)<br>• Efficient sintering activity (in terms of efficient reduction of metal powders surface oxides)<br>• High mechanical strength of the sintered parts<br>• Smooth and defect-free surfaces of the sintered parts<br>• High consistency of powders and sintered parts' properties (in-lot and lot-to-lot) | • High consistency, tight specification of key properties:<br>– Ash<br>– Moisture<br>– Particle size<br>– Crystallinity<br>• Defined raw material and process for synthetic graphite<br>• Full control of the supply chain for the natural graphite: from the mine, through the processing, to the customer.<br>• Due to its high reactivity synthetic graphite is the optimal solution to improve the density of the final part<br>• Good compressibility in blends with iron – low spring back<br>• High diffusion rate and reactivity with Fe |

We propose that a tailored selection of Graphite can effectively influence the 4 P's mix of PM parts production.

# Graphite selection for powder mixes properties

Graphite, Primary Synthetic or Natural, with d90 in the range of 10μm-44μm, has no significant impact on PM mixes' flowability [1].

| GRAPHITE TYPE | APPROX. D90 [μm] | A HALL FLOW RATE (s/50 g) | B HALL FLOW RATE (s/50 g) |
|---|---|---|---|
| Natural A | 10 | 32 | 34 |
| Natural B | 25 | – | 36 |
| F10 | 10 | 33 | 35 |
| PG10 | 10 | 33 | 35 |
| PG25 | 25 | 34 | 36 |
| F25 | 25 | 33 | 34 |
| KS44 | 44 | 33 | – |
| PG44 | 44 | 33 | – |

Hall Flow Rate of several Powder Mixes containing Synthetic and Natural Graphites of three different particles size distributions: 10μm, 25μm, 44μm are the d90 values.
A: ANCORSTEEL B +0.65%C +2%Cu +0.8%wax. [courtesy of Hoeganaes Corporation Europe].
B: ATOMET DB46 +0.6%C +0.6%wax [courtesy of QMP - Rio Tinto Powders]

Consistent, fast flowability is connected to PM parts weight stability. Slight gain in weight standard deviation (8 to 9%) when shifting from 10μm to 25μm d90 Natural Graphite has been reported [1, Application Case 1].
In order to prevent the risk of fine powders dusting, it is typically recommended to limit the use of Graphite powders with d90 lower than 10μm to bonded mixes only [2, 3].

# Graphite selection for improved PM sintering process

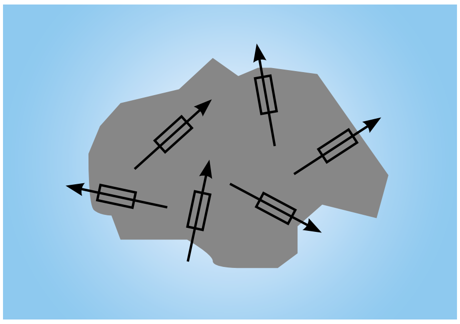
Graphite plays a fundamental role in PM parts sintering process. Graphite powder particles dissolve in iron-based PM steel matrix if the system is above $\alpha \rightarrow \gamma$ transformation temperature and reduction of the iron oxide layer, covering powder particles surface, has taken place.

Formation of inter-particles sinter necks begins after reduction of the surface iron oxide layer. Significantly enhanced oxides reduction activity has been reported for primary synthetic graphite, compared to natural flakes of similar particles size distribution [1, 4, 5, 6, 7, 8].

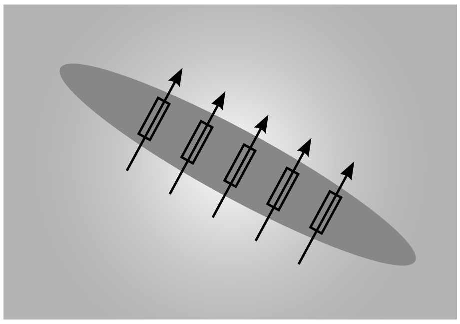
Primary synthetic graphite presents smaller, isotropically oriented crystallites, compared to natural graphite of similar particles size distribution [1].

Benefits in the sintering process from using primary synthetic graphite TIMREX® F10, F25, KS44 instead of natural flakes of respective particles size distribution can be summarized as:

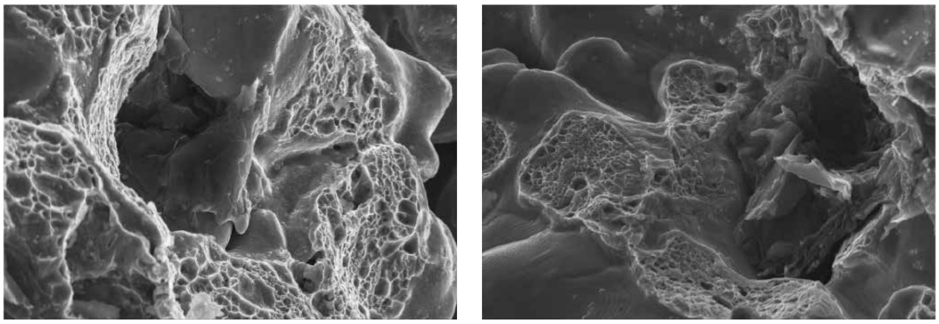
- earlier and more efficient iron-based powders oxide layer reduction [4, 5, 6, 7, 8]
- prolonged effective sintering time for better necks formation or shorter sintering time [4, 5, 6, 7, 8]
- earlier for carbon diffusion, resulting in steeper Copper concentration gradient in iron-based powders and stronger, Cu-richer sintering necks in FeCuC mixes [8; see also 6, 7]
- consequently: higher alloyed carbon in sintered PM parts, lower dimensional change sintered-to-die, slightly higher mechanical performance [1, 6, 7, 8].



Primary synthetic graphite



Natural graphite



1µm Mag = 10.00 K X EHT = 15.00 kV

2µm Mag = 10.00 K X EHT = 15.00 kV

Fracture surface of PM compacts utilizing natural graphite PG10 (to the left) and primary synthetic graphite F10 (to the right) in a Höganäs AB AstaloyCrM+0.5%C mix. Heating performed in dilatometer in 90%$N_2$/10%$H_2$ atmosphere to 1120°C. The earlier formation of sintering necks allowed by primary synthetic graphite F10, compared to natural flakes of the same particles size distribution is confirmed by finer dimples fracture in sinter necks fracture surfaces [5 - by Chalmers University, Sweden].

Clear indication of benefits for pre-sintering stage have also been shown by ENSACO® 250G carbon black, capable in a narrow temperature range to boost oxides reduction [5]. Since 2012, several publications have been covering the collaboration of IMERYS Graphite and Carbon with Chalmers University (Sweden).

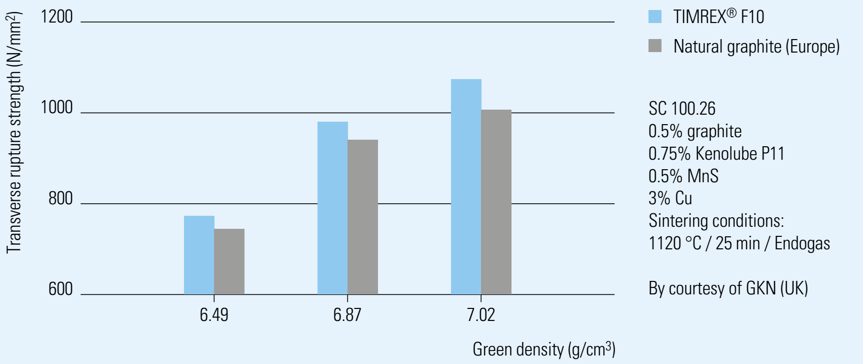
# Graphite selection for improved mechanical performance

Different concentration of alloyed Carbon in sintered PM parts is observed, when different graphite grades are used in powder mixes based on both Atomized and Sponge Iron, as well as Diffusion-bonded powders. Higher Hardness, Tensile Strength are direct consequences of higher level of alloyed Carbon, achieved by using TIMREX® F10, F25, KS44 instead of natural flakes of respectively the same particles size distribution.
Same trend is observed for sintering both in endogas and in 90/10 $N_2/H_2$ atmosphere [1].

| FE / GRAPHITE MIXTURE | CONCENTRATION OF ALLOYED CARBON | |
|---|---|---|
| | TIMREX® F10 | NATURAL GRAPHITE (EUROPE) |
| **ASC 100.29**<br>+0.8% Graphite<br>+ 0.8% Zn-stearate | 0.78 | 0.74 |
| **ASC 100.29**<br>+0.8% Graphite<br>+ 0.8% Zn-stearate<br>+ 2.0% Cu | 0.78 | 0.72 |
| **NC 100.24**<br>+0.8% Graphite<br>+ 0.8% Zn-stearate | 0.67 | 0.63 |
| **DYSTALLOY AE**<br>+ 0.6% Graphite<br>+ 0.5% Zn-stearate | 0.53 | 0.50 |

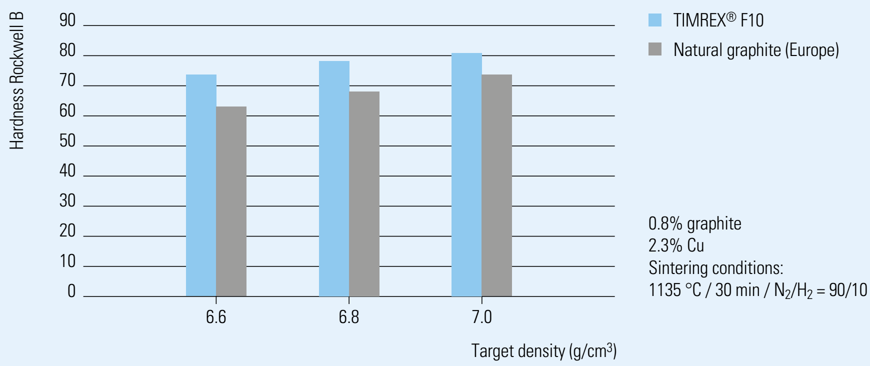
The ASC- and NC-powders were prepared as STARMIX
Sintering conditions: 1120°C / 30 min / $N_2/H_2$ / 90/10

By courtesy of Höganäs AB




SC 100.26
0.5% graphite
0.75% Kenolube P11
0.5% MnS
3% Cu
Sintering conditions:
1120 °C / 25 min / Endogas

By courtesy of GKN (UK)




0.8% graphite
2.3% Cu
Sintering conditions:
1135 °C / 30 min / $N_2/H_2$ = 90/10

# Graphite selection for reduced dimensional change

This is the workhorse of our primary synthetic graphite powders, the materials that have advanced powder metallurgy into the modern age of high demands PM parts.

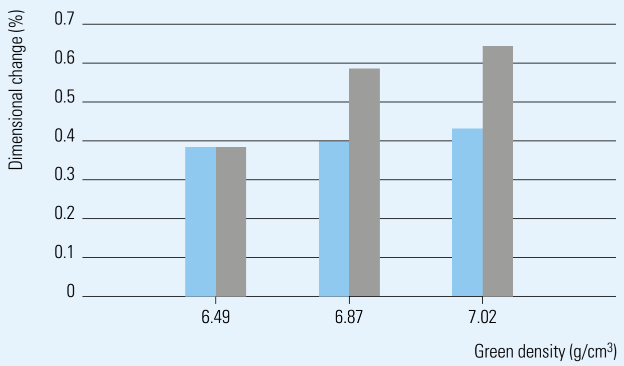
## DIMENSIONAL CHANGE AND ITS STANDARD DEVIATION AS A QUALITY PARAMETER

The high reproducibility of sintered dimensions results in enhanced quality of PM parts production. Possible cost reductions due to less sizing, machining, out of specs in lot-to-lot inspections are also to be considered.

| GRAPHITE | SINTERED DENSITY [g/cm³] | DIMENSIONAL CHANGE | |
|---|---|---|---|
| | | ∆l [%] | Standard deviation |
| TIMREX® F10 | 7.11 | 0.03 | 0.008 |
| NATURAL GRAPHITE (EUROPE) | 7.13 | 0.03 | 0.018 |

ASC 100.29 / 0.8% graphite / 0.8% Zn-stearate (STARMIX)
Number of investigated parts: 2000
Sintering conditions: 1120°C / 30 min / $N_2/H_2$ / 90/10

By courtesy of Höganäs AB




TIMREX® F10
Natural graphite (Europe)

SC 100.26
0.5% graphite
0.75% Kenolube P11
0.5% MnS
3% Cu
Sintering conditions:
1120 °C / 25 min / Endogas

By courtesy of GKN (UK)

# TIMREX® graphite and ENSACO® carbon black for powder metallurgy and hard metals

| | | Ash [%] | Crystallite height Lc [nm] | Scott density [g/cm³] | Particle size distribution d50 [µm] (*) Vibrated sieving | d90 [µm] (*) Vibrated sieving |
|---|---|---|---|---|---|---|
| TIMREX® synthetic graphite | **PM special** | | | | | |
| | F10 | < 0.6 | 80 | 0.09 | 6.8 | 12.6 |
| | F25 | < 0.6 | > 90 | 0.14 | 11.0 | 27.2 |
| | **KS graphite** | | | | | |
| | KS4 | 0.07 | 50 | 0.07 | 2.4 | 4.7 |
| | KS6 | 0.06 | 60 | 0.07 | 3.4 | 6.5 |
| | KS10 | 0.06 | 70 | 0.09 | 6.2 | 12.5 |
| | KS15 | 0.05 | 90 | 0.10 | 8.0 | 17.2 |
| | KS44 | 0.06 | > 100 | 0.19 | 18.6 | 45.4 |
| | KS75 | 0.07 | > 100 | 0.24 | 23.1 | 55.8 |
| | KS5-75 TT | 0.04 | > 100 | 0.41 | 38.8 | 70.0 |
| | KS150 | 0.06 | > 100 | 0.42 | 40% > 63 µm (*) | 20% > 100 µm (*) |
| | KS150-600 SP | 0.06 | > 100 | 0.67 | 83% > 250 µm (*) | 22% > 500 µm (*) |
| TIMREX® natural graphite | **PM special** | | | | | |
| | PG10 | 3-4 | > 100 | 0.06 | 6.4 | 12.5 |
| | PG25 | 3-4 | > 200 | 0.07 | 10 | 22 |
| | PG44 | 3-4 | > 200 | 0.10 | 22.4 | 49.6 |
| | **FR graphite** | | | | | |
| | -100 mesh FR | < 7 | > 350 | 0.75 | 50% > 75 µm (*) | 7% > 150 µm (*) |
| | 50x100 mesh FR | < 7 | > 350 | 0.78 | 68% > 180 µm (*) | 10% > 300 µm (*) |

| | | Ash [%] | Moisture (as packed) [%] | Sulphur [%] | Pour Density [kg/m³] | BET Nitrogen Surface Area [m²/g] |
|---|---|---|---|---|---|---|
| CARBON BLACK | | | | | | |
| | ENSACO® 150G | 0.01 | 0.1 | 0.01 | 190 | 50 |
| | ENSACO® 250G | 0.01 | 0.1 | 0.01 | 170 | 65 |

● Recommended
○ Especially Recommended

## APPLICATIONS AND RECOMMENDED GRADES

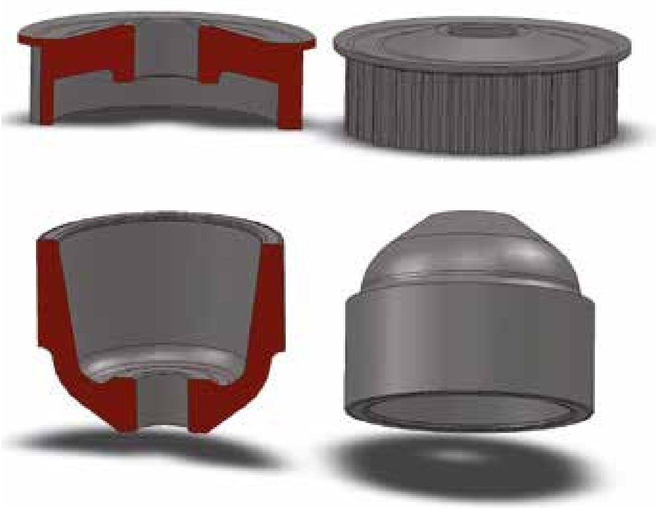
| Special alloys [Al, Mg, Ti] | Hard metals [WC, TiC, mixed carbides] | HSS | PIM/ MIM | Fe-sintered engineering parts | Fe-self lubricating engineering parts | Diamond tools | Copper/ bronze bearings | Copper friction parts copper clutch facings | Grade | Group |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | **PM special** | TIMREX® synthetic graphite |
| | | | | ○ | ○ | ● | ● | | F10 | TIMREX® synthetic graphite |
| | | | | ○ | ○ | ● | ● | | F25 | TIMREX® synthetic graphite |
| | | | | | | | | | **KS graphite** | TIMREX® synthetic graphite |
| ○ | ● | ● | ● | ● | | | | | KS4 | TIMREX® synthetic graphite |
| ○ | ● | ○ | ○ | ● | | | | | KS6 | TIMREX® synthetic graphite |
| ● | ● | | | ● | ● | ● | ● | | KS10 | TIMREX® synthetic graphite |
| ● | ○ | | | ● | ● | ● | ● | ○ | KS15 | TIMREX® synthetic graphite |
| ● | ● | ○ | | ● | ○ | ● | ● | | KS44 | TIMREX® synthetic graphite |
| | | | | | ○ | ○ | ● | ● | KS75 | TIMREX® synthetic graphite |
| | | | | | ○ | ○ | ○ | ● | KS5-75 TT | TIMREX® synthetic graphite |
| | | | | | | | ● | ○ | KS150 | TIMREX® synthetic graphite |
| | | | | | | | | ○ | KS150-600 SP | TIMREX® synthetic graphite |
| | | | | | | | | | **PM special** | TIMREX® natural graphite |
| | | | | ○ | ● | | | | PG10 | TIMREX® natural graphite |
| | | | | ○ | ○ | ● | | | PG25 | TIMREX® natural graphite |
| | | | | ○ | ○ | ● | ● | | PG44 | TIMREX® natural graphite |
| | | | | | | | | | **FR graphite** | TIMREX® natural graphite |
| | | | | | ● | ○ | ● | ○ | -100 mesh FR | TIMREX® natural graphite |
| | | | | | | | ● | ○ | 50x100 mesh FR | TIMREX® natural graphite |
| | | | | | | | | | | |
| | | | | | | | | | | CARBON BLACK |
| | ○ | | | ● | | | | | ENSACO® 150G | CARBON BLACK |
| | ○ | | | ● | | | | | ENSACO® 250G | CARBON BLACK |

## CASE 1: MEDIUM-LOW DENSITY PARTS

Optimized production of complex shape PM parts, like water pump pulleys or ABS sensor rings has been recently discussed in literature [1, 2, 3, 8, 9]. Such components are typically based on FeCuC mixes, at sintered density levels below <6.7g/cm³.
Sintering is typically run at 1120°C for 30min or even shorter time. Component's main requirement is dimensional stability (inner diameter). Key-feature requested to PM mixes is consistent and possibly enhanced flowability, in order to reduce weight scatter and possibly increase productivity. The selection of graphite powder for such applications can focus on one main aspect: at low density levels a shift from 10µm-d90 to larger particles size distributions (25 or 44µm d90) can be rather easily managed in compaction set-up.
Production trials have been run on two water pump pulleys (one with geared flank). Servo-Hydraulic presses were utilized (Dorst TPA250/3HP and Dorst TPA160/3HP), with bag-on-press system, in order to optimize mass flow. Gravity filling method was utilized. Presses were equipped with weight and compaction force measurement on every individual compacted part. About 8000 parts per each component were produced. The table summarizes the effective reduction of weight standard deviation obtained simply by shifting from 10µm to 25µm d90 graphite powder in the mix.

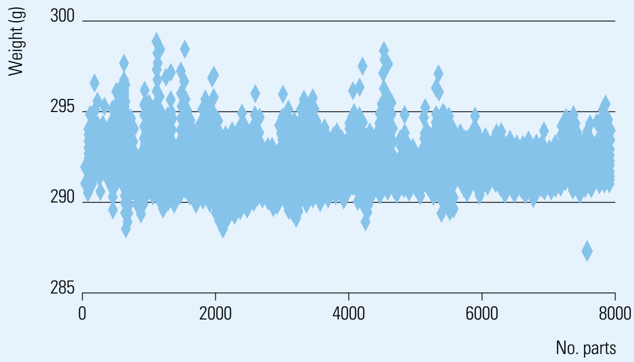
| **COMPONENT CODE** | **GRAPHITE TYPE** | **TYPE** | **APPROX. D90** [µm] | **AVERAGE PART'S WEIGHT** [g] | **STANDARD DEVIATION** [g] |
|---|---|---|---|---|---|
| P1 | PG25 | Natural | 25 | 291.91 | 1.14 |
| P1 | Natural A | Natural | 10 | 292.92 | 1.25 |
| P2 | PG25 | Natural | 25 | 295.00 | 0.77 |
| P2 | Natural A | Natural | 10 | 294.91 | 0.96 |

Weight Stability of pressed Water Pump Pulleys. Over 8000 parts production trials. Powder mixes consisting of Fe +1.5% Cu +0.65% C +0.8% Lubricant. Selection of Natural Graphites with two particles size distributions: 10µm and 25µm d90.

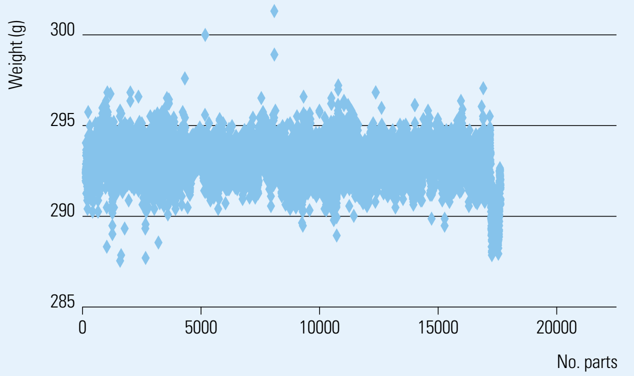


Water pump pulleys type P1 (above) and P2 (below).

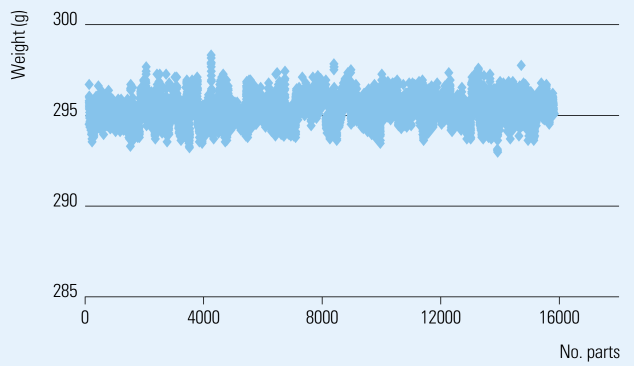
The suggested choice for similar medium-low density PM parts is natural graphite TIMREX® PG25 (25µm-d90). For components requiring particularly tight dimensional specifi cations, the recommended choice is primary synthetic graphite TIMREX® F25 (25µm-d90).



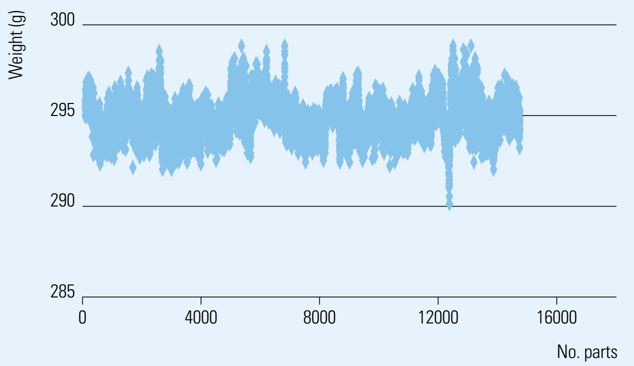

Weight Scatter Water Pump Pulleys **P1**
Green Density 6.7g/$cm^3$.
**FeCuC mix with PG25 graphite**




Weight scatter water pump pulleys **P1**
Green density 6.7 g/$cm^3$.
**FeCuC mix with graphite natural A**




Weight scatter water pump pulleys **P2**
Green density 6.7 g/$cm^3$.
**FeCuC mix with PG25 graphite**




Weight scatter water pump pulleys **P2**
Green density 6.7 g/$cm^3$.
**FeCuC mix with graphite natural A**

# Application cases

### CASE 2: VALVE GUIDES

Typical example of valve guide.

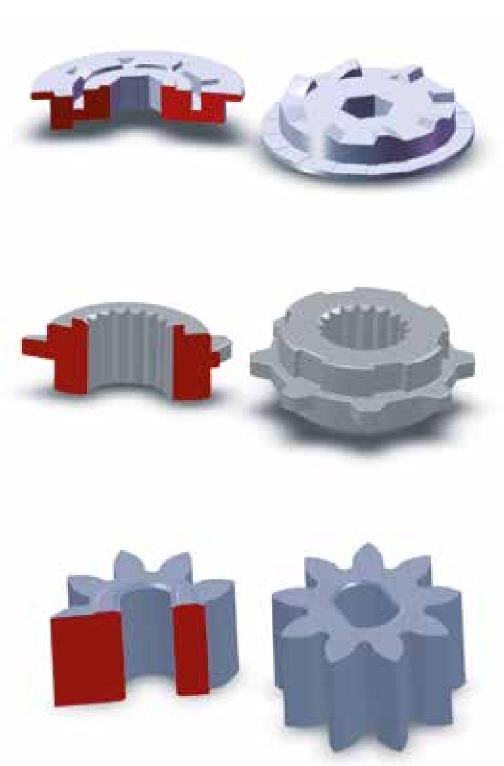
In year 2000 a publication by Kanezaki [10] benchmarked Cast Iron and PM Valve Guides (Fig. 2) with regards to durability –i.e. wear resistance.

Kanezaki indicates for such components a reference- chemical composition of Fe +1-6%Cu-P +0- 0.4%Mo +1-3%C. Sintered density is in general below 6.8g/cm3. Sintering is typically run at 1050-1120°C for 30min. Graphite plays double role in this application: Iron matrix hardening as well as friction coefficient modifier, by solid lubrication. The first function is achieved by efficient diffusion into the original Iron powder particles. It must be pointed out that such components are usually machined after sintering and consequently a certain level of mechanical resistance must be achieved – typically Pearlite is desired as dominant microstructure. Solid lubrication is instead obtained by nondiffused Graphite particles that remain within the pores of the microstructure. Selection of Graphite powder for this application typically consists of splitting the total required Carbon in two selected Graphite powders. Typically a Primary Synthetic or Natural graphite (10µmd90) in the range of 0.5-0.8% is meant to diffuse and reinforce the Iron matrix and a coarser Graphite powder (44µm d90) is meant to work as solid lubricant.

### CASE 3: HIGH DENSITY/HIGH PERFORMANCE PARTS

General indications can be given for the selection of optimal graphite powder for high performance/high precision PM parts:

- due to higher reactivity during sintering, primary synthetic graphite TIMREX® F10, F25, KS44 are the preferred choice when sintering activity and hardenability need to be boosted: this is the case for Cr-alloyed powders, sinter-hardening parts, structural components like con-rods and gears [1, 5, 6, 7, 11].
- when the desired performance is Dimensional Stability (for instance when weight classes are established for a given PM part production), primary synthetic graphite like TIMREX® F10, F25, KS44 contribute to reproducibility of dimensional change values [1, 6].
- earlier start of sintering process thanks to TIMREX® F10, versus natural flakes of similar particles size distribution [5], suggest that sintered cracks or residual tensions in complex-shape PM parts might be reduced by selecting primary synthetic graphite.
- for higher density parts 10µm-d90 is the suggested particles size. Finer particles size distributions are suggested only in combination of bonding treatments.



# Conclusions

| KEY REQUIREMENTS | GRAPHITE TYPE SELECTION |
|---|---|
| high mechanical performance<br>high dimensional stability<br>sinter-hardened, Chromium-based<br>PM steels<br>complex shape PM parts<br>valve guides/seats | TIMREX® KS4<br>TIMREX® F10<br>TIMREX® F25<br>TIMREX® KS44 |
| Reduction of PM parts cost<br>(by higher productivity,<br>lower raw-material cost) | coarser natural flakes:<br>TIMREX® PG25<br>TIMREX® PG44 |

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## ACKNOWLEDGMENTS (IN ALPHABETIC ORDER)

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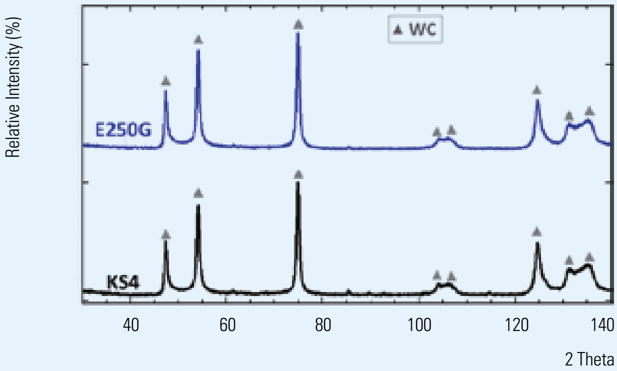
# TIMREX® graphite and ENSACO® carbon black for hard metals

**GRAPHITE AND CARBON BLACK POWDERS FOR HARD METALS**

IMERYS Graphite & Carbon has a long term presence in Hard Metals market as supplier of high purity, high consistency Primary Synthetic Graphite Powders [1]. TIMREX fine grades, like KS4, KS6, KS15, can be offered with tailored specifications on maximum levels of impurities like Sulphur, Calcium, Silicon, Iron, that are detrimental for Hard Metals manufacturing [2, 3]. In addition to graphite, we also offer high purity carbon black with high BET. The high reactivity of ENSACO carbon black make these products particularly suitable for the synthesis of nano-sized WC powders starting from tungsten oxide [4, 5, 6, 7].

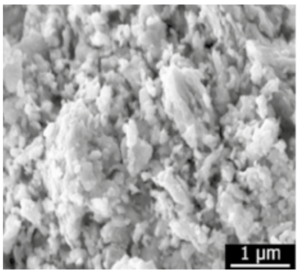
Tungsten metal powder (W) and Tungsten oxide powder ($WO_3$) have been mixed with different carbon powders (E250G and N991 carbon blacks, KS4 and KS44 graphites) for 2 hours at 300 rpm in a Fritsch Pulverisette planetary mill. Carburization has been performed in a Netzsch DIL402C dilatometer [5,6,7].

**WC produced from metallic tungsten powder (W)**

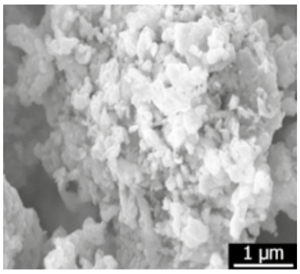



1100°C – 30 min, Ar

XRD patterns of WC powders obtained after carburizing W+C mixes in Ar at 1100°C




KS4



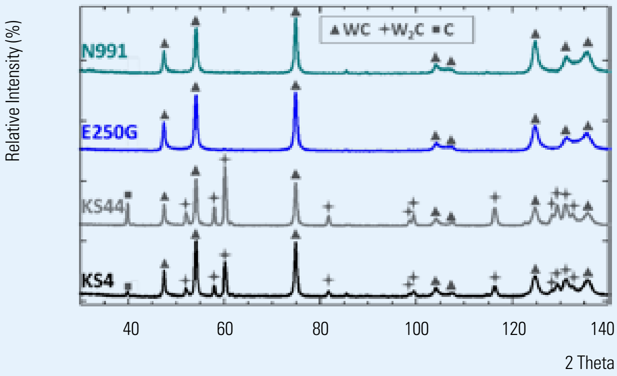

E250G

SEM pictures of WC powders obtained after carburizing W+C mixes in Ar at 1100°C.

- Inert atmospheres are recommendable for the synthesis of WC when metal W powders are used as precursors. In these conditions, fine WC powders can be obtained at 1100 °C using either graphite or carbon black powders.
- The resulting WC powders consist of agglomerates of submicron particles with irregular platelet morphology.

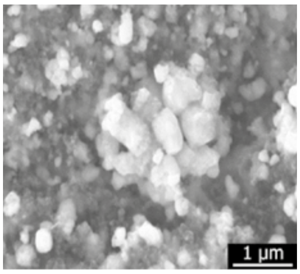
| WC POWDERS | S [ppm] | BET [m²/g] | PARTICLE SIZE [nm] |
|---|---|---|---|
| KS4 | 31 | 2.67 | 144 |
| E250G | 20 | 2.55 | 151 |

## WC produced from tungsten oxide powder ($WO_3$)

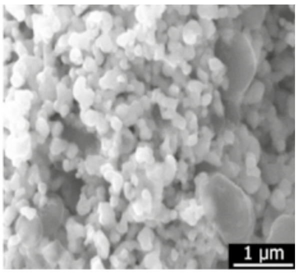



1100°C – 30 min, Ar-50$H_2$

XRD patterns of WC powders obtained after carburizing $WO_3$+C mixes in Ar-50%$H_2$ at 1100°C




E250




N991

SEM pictures of WC powders obtained after carburizing W+C mixes in Ar at 1100°C.

- It is possible to synthesize WC directly from $WO_3$ powders. In this case, atmospheres containing hydrogen are needed to activate reduction of oxides at lower temperatures, whereas at higher temperatures reduction is promoted by the presence of carbon.
- Carburization reaction takes place at lower temperatures for carbon black (E250G < N991) compared to graphite.
- Carburization in Ar–50%$H_2$ of mixes containing $WO_3$+Carbon black is complete at 1100°C , whereas for $WO_3$+graphite powders complete transformation to WC is achieved at higher temperatures (1300°C).
- The resulting WC powder have spherical morphology, sub-micron particle size and crystalline grain sizes below 30 nm (estimated by XRD).
- The BET surface area is higher compared to WC powders obtained by metallic W. In particular, E250G gives much higher BET values compared to N991, indicating a finer grain size.

| WC POWDERS | S [ppm] | BET [$m^2/g$] | PARTICLE SIZE [nm] |
|---|---|---|---|
| N991 | 22 | 2.92 | 131 |
| E250G | 18 | 6.90 | 56 |

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