

## CEMENTED CARBIDE LINER SPECIFICATION

### 1. INTRODUCTION

Forced cemented carbide liners are used in hyper compressors for the production of Low Density Poly Ethylene (LDPE). At extreme working conditions, up to 3500 bar, and with extreme run times these liners must be produced from the highest quality cemented carbide material. TRIBO Hartstoff uses selected raw materials of exactly the correct specification and modern manufacturing techniques during the production and finishing of their components.

Specially developed test procedures are used both during and after production.

This specification covers cemented carbide, chemical, physical, and metallurgic properties, the production process, testing, and the certification procedure as well as a description of our intermediate and final packing methods.

### 2. NORMS/WORKING SPECIFICATIONS

This specification is based on the following norms and working specifications. In each case the last version is valid.

ISO 3369, ISO3878, ISO3326, ISO4505, ISO 3327, ISO 3312, ISO 3878, ISO 4505, ISO 4506, ISO 4499; TÜV-MPA-HM-WS 911; TRIBO Hartstoff working specifications: LCH 009/014/103/104, LCW 002/004, QF 006, QM001/002/019-024

### 3. MATERIAL PHYSICAL PROPERTIES

The most important parameters are listed below:

Parameter	H20	V20	Units
a) Co Concentration	6,0	8,5	%
b) TC Concentration	94,0	91,5	%
c) Grain Size TC (FSSS)	1,3	2,5	µm
d) Density (ISO 3369)	14,85 ± 0,1	14,60 ± 0,1	g/cm <sup>3</sup>
e) Hardness	1590 ± 30 92,0 ± 0.2	1400 ± 30 90,3 ± 0.2	HV30 (ISO 3878) HRA
f) Transversal Rupture Strength (ISO 3327) measured: round bar	≥ 3410 ≥ 495 x 10 <sup>3</sup>	≥ 3550 ≥ 515 x 10 <sup>3</sup>	MPa PSI
g) Young modulus (ISO 3312)	625	600	GPa
h) Compressive Strength (ISO 4506)	6380 925 x 10 <sup>3</sup>	5160820 748 x 10 <sup>3</sup>	MPa PSI
i) Poisson's Ratio	0,21	0,20	
j) Relative Impact Resistance	40	45	
k) Endurance Limit, Rotating Beam 10 <sup>8</sup> cycles	960	870	MPa
l) Relative Wear	173	109	1 /Lost Vol.

Parameter	H20	V20	Units
m) Tensile Strength	1920	1750	MPa
n) Fracture Toughness	9,6	9,2	MPa•m <sup>½</sup>
o) Thermal expansion coefficient	5,4x10 <sup>-6</sup>	5,8x10 <sup>-6</sup>	K <sup>-1</sup>
p) Thermal Conductivity Coefficient	116	114	W•m <sup>-1</sup> •K <sup>-1</sup>
q) Coercivity (ISO 3326)	18,3 ± 2,4 229,8 ± 30.1	11,2 ± 2,4 140,7 ± 30.1	kA/m Oe
r) Specific Magnetic Saturation	10,9 ± 0,5	15,3 ± 0,8	μTm <sup>3</sup> /kg.
s) Porosity (ISO 4505)	≤A02 /B00 /C00	≤A02 /B00 /C00	
t) Graphite (ISO 4505)	No visible graphite (100x/200x).		
u) Eta - Phase (ISO 4499)	No Eta-Phase (100x/1500x).		

#### 4. PRODUCTION PROCESS

##### 4.1 RAW MATERIALS

Detailed raw material characteristics and requirements are provided to our suppliers. The specifications of our tungsten carbide and cobalt raw materials include all the important parameters to produce high quality cemented carbide with lowest level of impurities.

For example:

##### Tungsten carbide:

- Carbon content
- Grain size distribution
- Fine grain portion: 0 - 1 μm
- Impurities/traces

(Detailed information under client request)

##### Cobalt powder:

- Oxygen content
- Grain size
- Impurities/traces

(Detailed information under client request)

##### 4.2 MIXTURES PRODUCTION

Both raw materials (TC and cobalt) are mixed in special attrition mills with milling liquid ethanol. The resulting suspension is wet sift in a narrow mesh sieve. Afterwards plasticization (paraffin addition) takes place and the final mixture is dried in a spray drying system. For this, TRIBO has two separate lines for the different mixtures, this is to avoid contamination. One line is used only for hard metal TC-Co powders used in the production of our high pressure parts.

- **Wet sift over a narrow mesh sieve**
- **Special paraffin content**
- **Mixture production under Clean-room conditions**
- **Exclusive usage of fresh TC and Co charges (no recycling powders)**



#### 4.3 PRECOMPACTION TECHNIQUE

TRIBO Hartstoff has developed a special pre-compaction technique to generate precise and optimal compression relations in the green parts (compressed cemented carbide powder before sintering). This important step guarantees powder uniformity before Cold Isostatic Press process (CIP) is used.

- **Special pre-compaction technique**
- **Optimized diameter reproduction**
- **Good linearity**

#### 4.4 CIP PROCESS

We compress the green parts in a Cold Isostatic Press process (CIP) at very high pressure. These green parts are produced from the previous pre-compacted powder with optimal solidity and a reproducible diameter reduction. We proceed to turn the green parts to its exact green dimensions.

- **Very good compaction through the optimized paraffin content**
- **Reproducibility in shrinkage through an exact CIP compaction process**
- **The green parts are produced in one step**

#### 4.5 SINTER HIP PROCESS

Overpressure sintering process takes place in a special designed Sinter-HIP-Furnace. This equipment makes it possible to dewax (eliminate the paraffin) from the cemented carbide green part by using hydrogen in a homogeneous way and without damaging the internal structure. Next a vacuum is generated inside the furnace and temperature is elevated until it reaches the sintering point. After the required sintering time, pressure is applied to the system by means of argon to carry out the HIP process which closes all the pores in the component. This method, one step Sintering-HIP-Process, ensures the best possible microstructure and excellent physical properties!

- **One step process**
- **Sinter-HIP at optimal sintering temperature**
- **No macro-porosity; micro-porosity <A02, B00**
- **Excellent physical parameters - transversal rupture strength & compression strength**

#### 4.6 FINISHING

First step in the final manufacturing process the surface is ground and honed to preform status. After finishing this process the liner is checked generally by the TÜV organization. Next step after testing is shrinkage of the liner inside of the steel body. After this the complete liner with steel body is ground and honed once again to final dimensions. Surface roughness in the hole -  $0.01 \mu\text{m} \leq \text{Ra} \leq 0,005 \mu\text{m}$  is the normal TRIBO-Standard.

- **Polishing of the surface area with a band polish machine**
- **Surface roughness adjustable to the customer specification**
- **Surface roughness of  $0.01 \mu\text{m} \leq \text{Ra} \leq 0,005 \mu\text{m}$  if desired**



#### 4.7 SHRINKAGE JOINT

That the cemented carbide liner withstands the working tensile loads, it is stabilized in a shrinkage joint together with a steel body. This shrinkage joint is in such way laid out that the cemented carbide liner also with the highest applied operating internal pressure has no tensile loads affect. The used steel is double forged and vacuum glow. The proof of the composition, physical properties and the manufacture procedure takes place via appropriate test certificates from the steel manufacturer. The examination of the shrinkage joint takes place by using ultrasonic procedure. All data of the shrinkage joint are documented.

- **Defined adding conditions for reaching of an optimized shrinkage joint**
- **Examination of the contraction seat by the TÜV organization**

#### 4.8 MARKING

The liners are marked according to the drawing specification. This process is made using an electrolytic technique. The method ensures no surface damage in the marked area. If no marking instructions are specified on the drawing or by our customer the following information will be inscribed:-

- a) Order number
- b) Liner number
- c) Zero mark
- d) TRIBO Logo

### 5. PRODUCT VALUATION DURING THE PRODUCTION PROCESS

Throughout the whole production process different tests take place in order to check and document the quality.

**5.1 Raw Materials:** Specification acceptance, Co/Oxygen-Concentration grain size, grain size distribution, impurities/traces, sample for storage

**5.2 Mixtures:** Chemical composition  
Co/impurities/traces/plastificator content, sample for storage

**5.3 Sintering test:**

1. Lot sample, dimensions: 16x12x8 (6) (3) mm/ Mono static and CIP samples
2. Large samples, dimensions: 20x20mm / approx. 70 mm length
3. Polishing sample, dimensions: 50 mm diameter/ approx.100 mm length
4. Special samples for transverse rupture strength and compression strength tests; samples for storage

**Samples 5.31** are made for each lot and valued together with the mono-static samples from each lot.

**Samples 5.32** are cut out of the liners green part and are sintered together with the liner in the same sintering cycle.

**Samples 5.33** are made for each carbide batch and submitted to the grinding, ultra sonic, Eddy current and surface finish tests.

**Samples 5.34** are made and checked for each liner to test the transverse rupture strength; for new liner carbide grades we record the compression strength; samples for storage are taken from the sample material at 5.32.

## 5.4 Tests

The following tests are carried out on the samples.

Parameter	5.31	5.32	5.33	5.34
Density (ISO 3369)	X	X	X	X
Macroporosity (ISO 4505)	X	X	X	
Microporosity (ISO 4505)	X	X	X	
Microstructure (ISO 4499)	X	X	X	
Free carbon (ISO 4505)	X	X	X	
Eta-Phase (ISO 4499)	X	X	X	
Coercivity (ISO 3326)	X	X	X	X
Magnetic Saturation	X	X	X	X
Hardness HV30 (ISO 3878)	X	X	X	X
Transverse rupture strength (ISO 3327)				X
Compression strength (ISO 4506)				X
Grinding & polishing test			X	
Surface roughness			X	
Ultrasonic test			X	
Surface crack test			X	
Visual surface test	X	X	X	

The test results are documented in **test reports** or **data certificates** (3.1 certification). This information is supplied to the customer as part of the documentation file.

## 6. FINAL TEST

### 6.1 Dimensions and Surface Roughness Test

The dimensions of the liner are verified according to the drawing in final inspection. This information is supplied to the customer as part of the documentation file.

### 6.2 TÜV-Nord MPA-Test

The finished liners are tested by the Association for Material Tests and Industrial Security TÜV-Nord MPA. This independent institution has all the necessary equipment to measure components for the high-pressure industry. The TÜV-Nord MPA carries out the following tests:-

- 1) ultrasonic test
- 2) surface crack test and
- 3) visual test for surface defects.

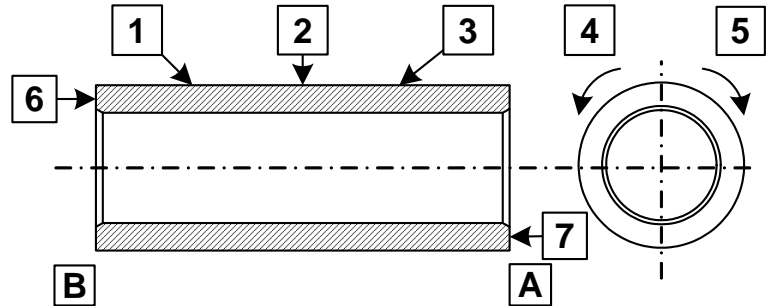
This information is supplied to the customer as part of the documentation file.



**6.21 Ultrasonic Test**

Beam direction: Position: 1 - 5  
Registration Range:  $Dq = 0,5 \text{ mm}$   
**Registration Range = Error Range**

Beam direction: Position: 6 - 7  
Registration Range:  $Dq = 2 \text{ mm}$   
**Registration Range = Error Range**



**6.22 Visual Test for Surface Defects**

The whole surface is analysed with 50 times' magnification.  
The registration ranges for surface defects are: Length/Width:  $0,015 \text{ mm}$   
Depth:  $0,005 \text{ mm}$

The defect classification is made according to maximum defect dimensions and form.

Surface roughness defects come under two classifications as follows: - :

**6.221 Surface defects due to the mechanical and grinding process**

Individual flat defects are permitted (depth  $< 0,01 \text{ mm}$ )

**6.222 Surface defects due to inhomogeneity inside the cemented carbide**

Defect Size	Defect Size Group	Maximal allowed defect number per group
$\geq 0,025 \text{ mm}$	$0,025-0,075 \text{ mm}$	5 Defects/1000 $\text{cm}^2$
$\geq 0,075 \text{ mm}$	$0,075-0,125 \text{ mm}$	3 Defects/1000 $\text{cm}^2$

All surface defects or material inhomogeneity has to be recorded.  
The minimum distance allowed between two surface defects of  $\geq 0,075 \text{ mm}$  must be more than 20 mm.  
To identify the position of any surface defects, the zero mark and the distance from the marking site of the liner are to be used as reference..  
The approval of defects larger than 0,125mm must be agreed between the supplier and the customer according to their position and quantity.

**6.23 Penetration Test**

For the penetration tests the same parameters are used as for the Visual Test 6.22.  
Registration Range =  $0,025 \text{ mm}$   
Any defect, which length is 3 times bigger than its width, is defined as linear. Linear defects are only permitted if due to the mechanical process.



## **7. FINAL PACKING**

In the final package TRIBO warrants that the liner will maintain its properties during transportation and a long term storage. To achieve this each liner is packed in several protective layers and finally in a sturdy wooden box suitable for fork-lift handling.

**7.1** The final packing must be carried out in a warm dry room and on a suitably clean table. The liner must be at room temperature to avoid the possibility of condensation forming on its surface. Plastic gloves over woollen hand gloves must be worn.

**7.2** The liner must be thoroughly cleaned with soft paper tissues and isopropyl alcohol. It must then be immediately covered with anticorrosive oil which must not contain sulphur or chlorine. Should it be necessary to clean the liner with a solvent it is very important to ensure that the solvent evaporation does not cause a temperature drop on the of the liner surface.

**7.3** The liner must then be wrapped in two layers of oilpaper and sealed with reinforced glass fibre tape. In hole of the Liver have to put 2 layers of oilpaper and then 2 or 3 bags of silica gel.

**7.4** The next level of our packaging for the Liner is one or two polyethylene layers (from 1 to 1,5 mm thick). The layers are fastened with contact band into three different points.

**7.5** The last layer of our special packaging is a polyethylene aluminium composite film with a thickness of 0,15mm, the coated side to the outside. This is then vacuum sealed.

**7.6** The liner is then placed into a wooden box using two slings. These slings remain inside the box to help with removal of the liner together with two pairs of woollen and surgical gloves.

**7.7** The box lid is then screwed down and the joints are sealed. Tension bands are placed around the box depending on the liner weight.

**7.8** On top of the box, besides our customer's address, our TRIBO Logo is painted and stickers with "Handle with care", "This side up" and "Protect against humidity" are glued.

**7.9** Information on special precautions for handling and using liners are added to the documentation package.



## 8. DOCUMENTATION

All test and inspection records are supplied to the customer as part of the documentation pack. In accordance with our Quality Control Department procedures, which are certified under DIN ISO 9001:2000, all samples as well as all recorded data are kept at TRIBO Hartstoff for at least 10 years.

The documentation package contains following documents:

- a) Liner material Certificate 3.1 according to DIN EN 10204:2005-01
- b) Surface Roughness & Measurement reports
- c) Ultrasonic, Eddy Current and Surface Quality (Visual & penetration) Tests Protocol
- d) Transverse Rupture Strength Test Protocol
- e) Special customer test documentation
- f) Liner handling instructions
- g) Test reports from other materials necessary to the liners (steel, etc.)

## 9. AGREEMENT

This specification is part of a quality system under DIN ISO 9001:2000 and is the property of TRIBO Hartstoff. It was developed as an informative guide for our customers. The partial or entire copying or delivery to a third party must not be made without the expressed written approval of TRIBO Hartstoff.

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