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TECHNICAL BULLETIN

Production of Metal Carbides using Thermax®

Thermax® N990/N991 Ultra Pure grade thermal carbon blacks, produced by Cancarb Limited, are well-known as the highest purity form of amorphous carbon available in both pellet (N990) and powder (N991) form. Thermax® carbon blacks are produced by the thermal decomposition of high purity methane and are characterized by very low surface area, very low ash and moisture content, and extremely low levels of sulphur and other impurities. These intrinsic properties have provided for wide acceptance in the metal carbide industry as the preferred carbon source for carburizing hard metals.

Hard metals, also known as cemented carbides, are a class of materials comprised of hard refractory metal carbide particles that are "cemented together" with a softer metallic binder, usually Co or Ni. As a result of their composition and microstructure, they have a good combination of hardness and toughness that makes them well suited for use as tools for metal cutting, metal forming, mining, rock drilling, road construction and many other similar applications.

The most common industrial carbide production process involves dry mixing precise amounts of refractory metal powders with either carbon black or graphite powders in a ball mill or high-energy mixer. These powder mixtures are placed in graphite containers and heated in a reducing atmosphere to temperatures between 1100°C and 2000°C to form refractory metal carbide powders.

The hard phase composition commonly consists of tungsten carbide (WC) particles having a specified average particle size, size distribution and carbon level. Several other carbides including TiC, TaC, NbC, Cr_3C_2, VC and ZrC rely on Thermax® N990/N991 Ultra Pure to achieve superior final properties. Additions of other carbide powders to WC carbides are sometimes used to enhance properties needed for specific applications. In comparison to graphite, Thermax N990/N991 Ultra Pure contains very low elemental impurities which may be detrimental to production and final product properties.

Table 1. Elemental impurities of carbon sources for metal carbide production

	Thermax® N990/N991 UP	Natural[1] Graphite	Synthetic[1,2,3] Graphite
Aluminum	<1	4	5.2
Antimony	<0.5		
Arsenic	<1	5	<0.03
Bismuth	<0.5		
Boron	<1		
Cadmium	<0.5		
Calcium	<2	43	
Chlorine	<6		
Chloride	<10		
Chromium	<0.5	1	<0.4
Copper	<1	15.1	1.2
Gallium	<0.5		
Iron	5	200	55.2
Lead	<1	0.5	
Lithium	<0.5	1	
Magnesium	<0.5		
Manganese	<0.5		0.14
Mercury	2		
Nickel	<0.5	33.7	2.9
Phosphorus	3		
Potassium	<1		
Silicon	20	130	
Sodium	10		
Sulphate	15		
Sulphur	<10		
Tin	<0.5		
Titanium	<0.5	28	
Vanadium	<0.5	10	
Zinc	<0.5		<0.8

All values in the table are expressed in parts per million (ppm)
Reported results are not comprehensive

[1] Ambrosi, A., Chua, C. K., Khezri, B., Sofer, Z., Webster, R. D., and Pumera, M. (2012). Chemically reduced graphene contains inherent metallic impurities present in parent natural and synthetic graphite. *Proceedings of the National Academy of Sciences of the United States of America*, *109*(32), 12899–12904.
[2] Maahs, H., Schryer, D.R. (1967). Chemical impurity data on selected artificial graphite with comments on the catalytic effect of impurities on oxidation state. *NASA Technical Note*
[3] Wong, C., Sofer, Z., Kubeova, M., Kuera, J., Matjkova, S. and Pumera, M. (2014). Synthetic routes contaminate graphene materials with a whole spectrum of unanticipated metallic elements. *Proceedings of the National Academy of Sciences*, 111(38), pp.13774-13779.

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The quality of finished carbide product varies depending on the process and notably on the quality of raw starting materials. High quality metal carbides require the best refractory powders to achieve consistent and favourable properties. It has been shown that the level of impurity in the finished carbide is determined entirely by the purity of the hard phase and the carbon source[4]. Elements like S, Ca, Na, P, Si and Al that are present in the raw materials remain in the finished WC powders, ultimately causing degraded hard metal properties. Thermax® N990/N991 Ultra Pure is the highest purity amorphous carbon achieved by using high quality natural gas feedstock.

Thermax® is produced from high quality methane feedstock in a flameless process operating at 1400°C. The process yields spherical/ellipsoidal carbon particles in the range of 240nm to 320nm. In the case of N990 Ultra Pure, soft pellets averaging 0.5mm in diameter that flow easily and create minimal dusting are produced in the process. The ash content specification for N990 and N991 Thermax® Ultra Pure grades is about 30% of current commercial synthetic graphite grades recommended for hard metal carburization[5]. The combination of these physical characteristics, low moisture content and chemical purity make Thermax® N990/N991 Ultra Pure the ideal choice for commercial production of refractory metal carbide powders that are required for modern hard metal applications.

Thermax® N990/N991 Ultra Pure products provide:

- Highest carbon purity, which enhances carbide yield and minimizes impurities in the final hard metal product.
- Lowest sulphur levels which allow carbide tool producers to manufacture premium micro-grained products.
- Lowest ash levels and low grit levels, minimizing the final metal carbide porosity and enhancing density.
- Nanometer scale particles that permit a reaction efficiency that cannot be matched by the coarser grain carbon sources such as graphite.
- Availability in pellet or powder form to conform to particular processing requirements.
- Soft and uniform pellets for ease of mixing, blending and for achieving optimal dispersion.
- High bulk density compared to other forms of carbon black.
- Very uniform lot-to-lot properties for demanding quality control requirements.

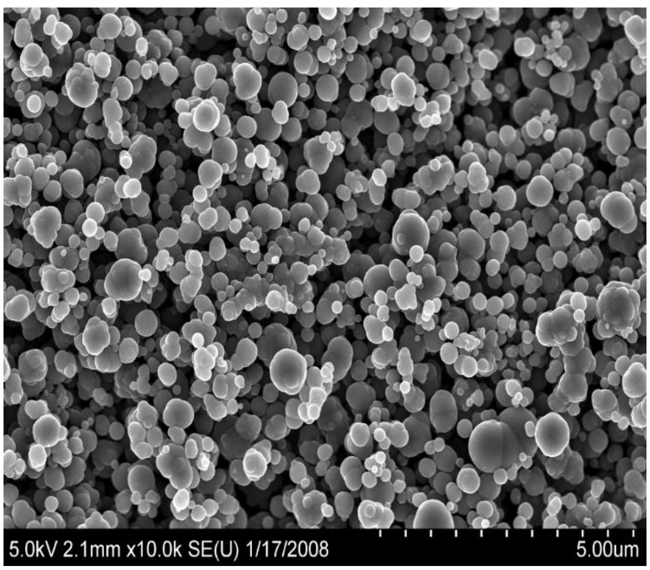

Figure 1. SEM imaging of N990/N991 Thermax® Ultra Pure showing spherical particle shape and approximate particle size

[4] Krstic, V. (1992). Production of Fine, High-Purity Beta Silicon Carbide Powders. *Journal of the American Ceramic Society*, 75(1), pp.170-174.
[5] Westinghouse Electric Corp (1975). *Silicon carbide coated graphite members and process for producing the same*. US3925577A.

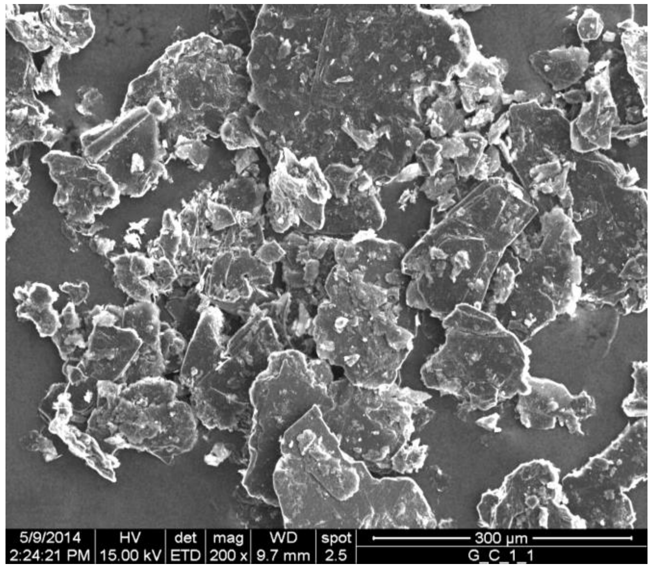

Figure 2. SEM imaging of graphite demonstrating particle shape

In the case of N990 Ultra Pure, soft pellets averaging 0.5 mm in diameter that flow easily and create minimal dusting are produced in the process. Thermax's® process produces particles ranging from 240-320 nm while industrial grade graphite is typically 10s of microns in length[6,7]. Thermax's® smaller particle size allows for a larger surface area for the reaction to occur, promoting faster carburization than would be achieved using graphite.

During the carburization reaction, many of the impurities present in the carbon and metal powders are removed from the WC powder via evaporation. Since the degree of purification increases with increasing carburizing temperature, finer WC powders typically have higher impurity levels than coarser powders produced from the same starting materials. Large-particle carbon black, Thermax® N990/N991 Ultra Pure is the largest particle size carbon black and is capable of producing metal carbides with extremely low impurity levels.

Table 2. Thermax® Ultra Pure specifications

ASTM Reference	Test Description	Thermax® N990 Ultra Pure
D1514	Sieve Residue	
	325 Mesh % (ppm), max.	0.0015 (15)
	Magnetics on 325 Mesh % (ppm), max	0.0005 (5)
D1506	Ash Content %, max	0.02
D6556	Nitrogen Surface Area, m^2/g	7.0-12.0
D1509	Heat Loss %, max	0.1
	Total Sulfur % (ppm), max.	0.006 (60)
D1508	Fines Content, as shipped, max %	8.0
D5230	Pellet Hardness (14 x 18 mesh)	
	Average, max	30
	High (average of 3 highest), max	50

[6] Phillips, C., Al-Ahmadi, A., Potts, SJ. et al. (2017). The effect of graphite and carbon black ratios on conductive ink performance. *Journal of the American Ceramic Society,* 52(16), pp.9520-9530.

[7] Rujijanagul, G., Jompruan, S., Chaipanich, A.,(2008). Influence of graphite particle size on electrical properties of modified PZT–polymer composites. *Current Applied Physics*, 8(3-4), pp.359-362.