COMPOUNDING OF SEMICONDUCTIVES FOR HIGH VOLTAGE CABLES

Colin G. RICHARDSON
Buss AG
4133 Pratteln
Switzerland
colin.richardson@busscorp.com

ABSTRACT

A key component of high, and very high voltage cable is the semiconductive layers; respectively the inner or conductor shield and the outer or insulation shield. The semiconductive extrudate must meet extremely well defined specifications with respect to pips or agglomerates and overall surface smoothness.

This presentation will detail the handling, feeding and compounding of conductive fillers into extrusion grade semiconductive compounds. It will compare the physical nature of these key raw materials and how this can lead to challenges in their incorporation in the polymer matrix.

Starting from stringently selected raw materials the quality determining step now lies in effective mixing of the ingredients, in particular the high level of pelleted carbon black. The energy input must be carefully controlled. The aim is to break down the carbon black to its primary aggregates and then to distribute the aggregates to the optimum degree (distributive mixing). An over mixing can lead to a decrease in conductivity due to undesirable break down of the primary aggregates.

Compounding techniques are therefore paramount in order to be able to meet these requirements.

KEYWORDS

Semiconductive, carbon black, compounding, dispersion, supersmooth.

INTRODUCTION

Both cable makers and end-user utilities are equally concerned about the expected life span of installed cable. Over the years significant improvements have been incorporated into cable design. Along with these technical advances greater demands are put on the performance of the cable. Cables today are installed in increasingly rough, hostile and costly (underground, undersea) environments.

In 1969 AEIC issued the first specification for polyethylene and XLPE insulated cable. Over the years this has been continually updated and new specifications introduced to ensure a very stringent and demanding set of criteria for power cable manufacture. Test requirements include physical, electrical and load cycling analyses. The semiconductive extrudate must meet extremely tight limits with respect to overall surface smoothness and the absence of lumps, pips or agglomerates against closely defined dimensions.

The quality of the semiconductive extrudate becomes of even greater importance as ever higher voltage rated cables are produced from polymeric compounds. The need for reduction of the insulation thickness to contend with the rising trend in voltage has been satisfactorily matched by improvements in cable manufacturing technology. The additional demands on the semiconductive are evident when typical thicknesses of conductor shield are compared with cable voltage, Table 1

Voltage (kV)	10	20	60	110	150	225	500
Shield (mm)	0.8	0.8	1.0	1.2	1.2	1.7	2.0

Table 1: Evolution of conductor shield thickness with voltage

Cable performance and cable integrity is dependent on all of the individual components that go to make up a cable. But specifically, and never more so as ever high voltage cables are produced from synthetic polymers, on the quality of the extrudate layers. These extrudate layers, predominantly applied in a single (triple extrusion) pass, are in intimate contact with each other and rely on zero interfacial imperfections for cable integrity.

Carbon black

Carbon black is one of the most versatile fillers used in plastics. It can be used to provide opacity, colour, UV protection, electrical conductivity, thermal conductivity and even reinforcement. In order to impart electrical properties to a polymer matrix typically carbon blacks from either the furnace process or the acetylene process are selected. Furnace blacks can be of petrochemical or carbochemical origin. Acetylene blacks, as the name implies, are produced from acetylene gas.

The smallest representative unit of carbon black is a particle. The properties of a given carbon black are related to the particle size and surface area of this primary unit. However carbon black does not exist as discrete particles. During the furnace manufacturing process the spherical carbon black particles permanently fuse together to form aggregates. A key property of the carbon black which controls its ultimate performance is the morphology of the primary aggregate. When many prime particles are fused together, usually with a high degree of branching or side chains, the resulting aggregate is described as having high structure. If the primary aggregate has relatively few prime particles the carbon black is described as having low structure, Fig1.
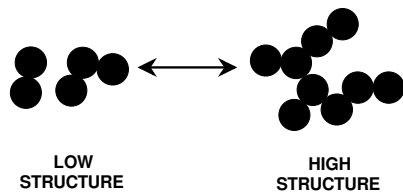
Fig 1: Schematic carbon black structure

The carbon black undergoes one further process step in order to convert the powder to a granule or pellet. The granulation reduces bulk and allows more convenient handling. However the crush strength of the granule plays an important role in the subsequent compounding step.

The structuring effect of the carbon black can have different even conflicting effects on its end use. Properties that are influenced by structure are summarised in Table 2.

High Structure	Property	Low Structure
Easier	Dispersibility	More difficult
Slower	Wetting	Faster
Higher	Conductivity	Lower
Higher	Viscosity	Lower
Lower	Loading capacity	Higher

Table 2: Principle effects of property and structure on compounding

Acetylene carbon black is the high temperature decomposition product of acetylene. It has the highest structure resulting in a very light, fluffy powder.
Depending on the process some furnace blacks can approach similar properties to acetylene black.

Conductivity and mixing

The conductivity imparted by carbon black is a function of loading level, the type of resin and the degree of dispersion. In certain end use applications it will also be affected by such things as temperature and the degree of mechanical stress applied to the product.

To achieve the conductivity levels, required in polyolefin based power cable shields and conductive extruded jackets, carbon black loadings typically of around 30 to 40% are needed. The highest degree of dispersion is mandatory to achieve an ultrasmooth surface quality, optimum and consistent electrical properties and the best in extrudability of the finished product (1).

The challenge now lies in incorporating the high level of carbon black combined with effective mixing. The energy input must be carefully controlled since the carbon black chains are sensitive and extremely prone to mechanical scission. Applied shear forces must be kept under control or electrical properties will be adversely affected due to shear breakdown of the carbon black. Fig 2 illustrates the negative result of over mixing
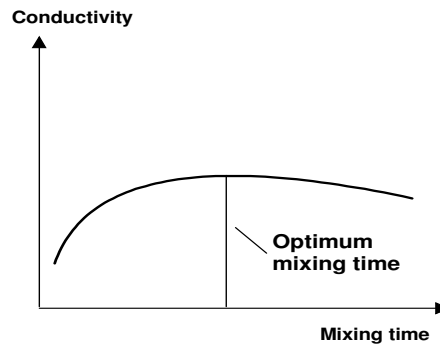

Fig 2: Effect of mixing on conductivity

Surface smoothness and cable construction

The modern HV cable construction is insulated with synthetic polymeric material and is composed of a central conductor, an insulating material, a ground or neutral layer and an outer jacket. Semiconductive layers are incorporated to eliminate electric field deformation and to provide a more uniform electric field within the insulating material, Fig. 3
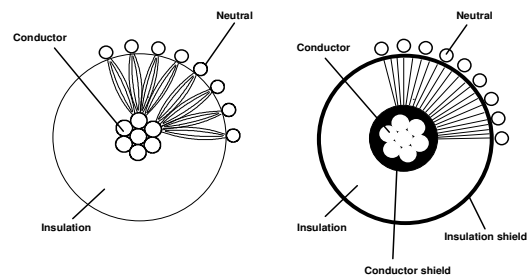

Fig 3: The role of the semiconductive layer in cable construction

It is now state of the art to co-extrude all three layers of the cable core in a single pass. This comprises the inner semiconductive layer (conductor shield), the insulation layer and the outer semiconductive layer (insulation shield). This trend has been driven by the considerable advantages exhibited by extruded shields, namely:
o More uniform electric field
o Higher resistance to corona
o Reduces damage to the insulation
o Smoother interfaces between insulation and semiconductive
o Minimises water tree growth

The production of high voltage power cables requires extreme care in avoiding contamination. The presence of foreign particles in the insulation layer can lead to serious consequences. Studies have confirmed that contaminants may initiate electrical or electrochemical processes which could lead to cable breakdown.
Similarly carbon black specifications have been significantly addressed with respect to ash content, sieve residue and even ionic content. Earlier work has reported the potential of ionic impurities, associated with different carbon black grades, to migrate into the insulation during service i.e. during electrical ageing (2).
Another major source of weakness is from imperfections at the conductor shield-insulation interface. Indeed the most

damaging behavior, eventually leading to cable breakdown, is due to protrusions from which electrical trees can initiate, Fig 4
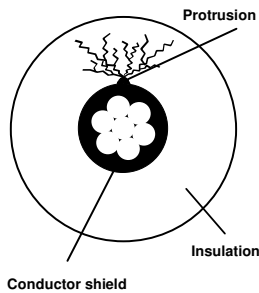

Fig 4: Schematic of tree growth from semiconductive protusion

Precautions to eliminate contamination and imperfections during cable manufacture take a predominant place in cable specifications. For example AEIC CS5, CS6 and CS7 state limitations on allowable protrusions into the insulation or conversely intrusions into the conductor shield, the severest limits being at the conductor shield - insulation interface, see Fig. 4. The German specification VDE 0273 appears to be even more stringent in this respect.

Protrusions in the semiconductive layer can arise for a number of reasons including impurities from carbon black, gel particles in the resin, pre-cure or scorch particles and mechanical damage during extrusion.
The routes to addressing these problems lie in raw material choice, specifications and processing techniques.
However, the ultimate in semiconductive dispersion is required to achieve proper surface smoothness, optimum and consistent electrical properties and the best in extrudability of the finished product.

The quality of the semiconductive extrudate becomes of even greater importance as ever higher voltage rated cables are produced from polymeric compounds. The need for reduction of the insulation thickness to contend with the rising trend in voltage has been satisfactorily matched by improvements in cable manufacturing technology. The additional demands on the semiconductive are evident when typical thicknesses of conductor shield are compared with cable voltage, see Table 1. A fifty fold increase in voltage is accomplished with a simple doubling of the semiconductive layer. The quality of the semiconductive ingredients and the sophistication of the compounding step are paramount for achieving this advance.

COMPOUNDING SEMICONDUCTIVES

Raw materials
The compounder will start with carbon black in the form of pellets. In the ideal mixing operation all carbon black pellets would be broken down into primary aggregates and then uniformly distributed throughout the polymer matrix. There are at least five key steps in the dispersive mixing process (3).
o Handling and feeding of powder
o Incorporation of powder in liquid

o Wetting of powder
o Separation of agglomerates
o Homogenisation

Handling and feeding of powder in this case means the carbon black. Physical properties of carbon black will themselves vary according to type. Three types of carbon black that have been studied include two furnace black grades and one from the acetylene process. They are referred to respectively as standard furnace black, extra clean furnace black and acetylene black. The physical properties are shown in Table 3

Carbon Black Type	N_2 Surface Area (M^2/g)	DBP Absorption (cc/100g)	Ash Content (ppm)
Furnace Black	160	114	120
Extra-Clean Furnace Black	50	160	30
Acetylene Black	70	225	< 1

Table 3: Physical properties of the chosen carbon blacks

One important characteristic not mentioned in this table is bulk density. The whole purpose of the blacks being pelletised is to provide a more easily transported and handled material. When bulk density is low it is extremely difficult to handle and feed. Indeed, if bulk density becomes too low it will be the rate determining step in the compounding operation by virtue of its difficulty to transport and introduce into the compounding machine.
Although a bulk density of >500 kg/m^3 is desirable it is more likey that ranges of 300 to 400 kg/m^3 can be experienced.

The carbon blacks were compounded into an ethylene ethylacrylate copolymer (EEA) having 18% EA content and melt index 6. Carbon black loadings from 34 to 41% were used from known conductivity curves.

Compounding system
The mixing and compounding was made on a MKS 70 Buss kneader reciprocating screw compounding line. The mixing chamber was of length 20l/d with the possibility of 3 feed inlets and a degassing dome, see Fig 5
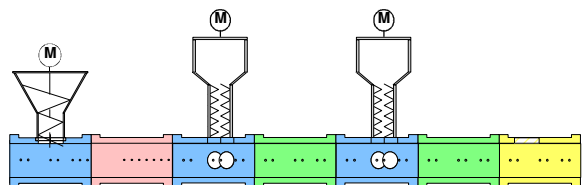

Fig 5: Kneader mixing chamber showing downstream feeding ports and degassing

It is standard practice to introduce the polymer(s) and stabilisers in the first port. The flexibility is now available for dividing the carbon black into the two downstream ports or even adding a proportion at the beginning along with the polymer. Other additives can be added at one of the three

position and liquids injected at the appropriate place via an injection nozzle.

The mixing theory of the Buss kneader has been well described elsewhere (4,5). The screw is designed with a discontinuous flight allowing three equidistant spaces or gaps per revolution. Three rows of fixed pins are mounted along the wall of the mixing chamber at correspondingly equidistant radial spacing. The kneader screw has a dual rotational and oscillating motion. Material in the kneader channels is intimately and repeatedly mixed between the flights of the moving screw and the stationary pins. At each interaction of a pin with a flight face product undergoes division, reorientation and recombination, see Fig 6.
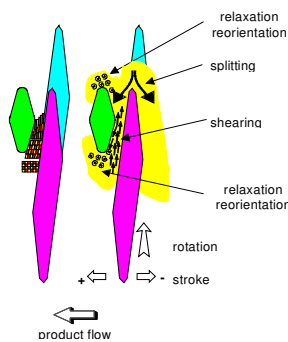

Fig 6: Interaction between flight and pin

As a result the stock undergoes repeated surface renewal during its progress along the kneader. For highly filled compounds this repeated action is extremely advantageous for wetting and filler incorporation.

The overall compounding line is complemented by a pressure generating pump followed, as required, by a screening device and pelletiser, see Fig 7.
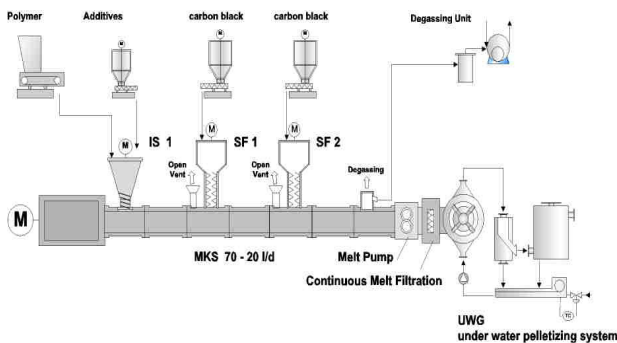

Fig 7: Semiconductive compounding line

The different zones of the kneader barrel can be heated or cooled using either oil or water tempering medium. The screw has a bored shaft allowing cooling water to circulate back and forth. The temperature of the melt pump is also controlled by circulating oil. Temperature is a key parameter in compounding semiconductives and the liquid tempering system has been shown to provide the most accurate control for these sensitive formulations.

Each step in the process has a specific role to play. Venting at the feed ports is needed for natural removal of entrained air; split feeding of the carbon for efficient material introduction, wetting and mixing under uniform and controlled shear whilst maintaining temperature below the degradation points of the polymers. Degassing if required may be effected with a vacuum pump for forced removal of air and other volatiles.

After the mixing process the material can then be screened (filtered) to add the final security of eliminating undispersed and undispersable hard particles followed by the final step of pelletising.
Additional pellet dryers can be installed according to the allowable moisture content and specifications on finished compound.

DISCUSSION

The parameters that have been evaluated include; carbon black type, carbon black loading, split feeding of carbon black, melt temperature, screw speed and dispersion. The laboratory work did not use a screening step, the goal being to have direct evaluation of the process on dispersion.

The compounded products were controlled first for carbon black content followed by important end use properties. Volume resistivity was used to assess the electrical properties or degree of conductivity. Viscosity and melt flow index were used to measure flowability. The level of dispersion was then evaluated from extruded tapes.

The dispersion degree had to be measured off line since automated scanning was not available in the compounding laboratory. Two tape tests were effected for a) tape rating and b) background dispersion. Tape quality rating is often used for compounds containing high carbon black loadings. This requires each sample to be extruded as a thin tape of constant thickness. For a given surface area each sample is evaluated under the microscope at x45 magnification and the number of "pips" or undispersed particles counted and classified according to size. From this a tape rating is assigned.

Other than the microscopic count of pips, an additional assessment was made of the so called "background dispersion". This is intended to evaluate the quality of the mixing operation at the micro level. These tapes were again visually tested under the microscope, compared against standards and rated "a" through "g". A summary of the data is given in Table 4.

All three carbon blacks were used at around the predetermined loadings to achieve volume resisitvity of <100ohm.cm^{-1} at ambient temperature. In general resistivity is seen to decrease for the different carbon blacks with increasing black content. Resistivity was also measured at the operating test temperature of 90°C. Now the values are higher and show wider differentiation. This is to be expected since the polymer matrix has changed from being partially crystalline at room temperature to an amorphous matrix at 90°C.

Carbon Black Type	Carbon Black Loading (%)	Feed Port(s)		Stock Temperature (^{o}C)			Process Parameters			
		SF 1	SF 2	T2	T5	T6	n rpm	P Kw	G Kg/hr	e-net Kwh/Kg
Extra Clean Furnace										
A1	41.0	25	75	118	138	112	200	5.9	15	0.317
A2	42.2	0	100	117	166	144	220	5.9	15	0.317
A3	43.3	25	75	132	183	173	300	7.5	22.5	0.230
Furnace										
B1	36.1	0	100	118	156	146	200	5.2	15	0.270
B2	36.2	25	75	132	167	160	250	6.6	20	0.255
B3	36.5	33	67	126	182	171	375	7.0	15	0.311
Acetylene Black										
C3	34.4	25	75	125	172	147	240	5.1	15	0.245
C1	37.3	0	100	113	169	142	220	4.4	10	0.311
C2	37.9	25	75	121	170	146	240	4.8	10	0.338

Table 4: Process data of different carbon blacks

Carbon Black Type	Loading	Volume Resistivity (ohm.cm)		Melt Flow (g/10min) Index	Viscosity (Pa.sec^{-1})	Tape Quality	
		Room Temp.	90°C			Pip count[1] 8 → 1	B.G.[2] a → g
Extra Clean Furnace							
A1	41.0	12.7	2724	30.9	3764	6	a
A2	42.2	5.9	117.5	20.1	4109	8	a
A3	43.3	4.7	400.9	12.6	4838	6	a
Furnace							
B1	36.1	20.4	2383.7	35.7	2978	3	c
B2	36.2	23.3	1806.1	33.6	2917	3	b
B3	36.5	31.6	6667.1	38.3	2803	4	c
Acetylene Black							
C3	34.4	198.1	67184.0	15.7	3886	7	c
C1	37.3	65.1	4262.3	5.6	4934	8	a
C2	37.9	72.2	1718.1	5.7	4873	8	a

Notes:(1) Pip count 8=excellent, 1= satisfactory
(2) a= super smooth, g= rough

Table 5: Product data of different carbon blacks

The anticipated conductivity increase with carbon black loading is observed in the case of the acetylene and extra clean blacks. The standard furnace grade actually showed a decrease in conductivity. This is attributed to the processing conditions where the screw speed was allowed to increase to the point of scission of the carbon black aggregates.

Electrical properties, mechanical properties and most importantly surface smoothness are the overriding criteria for HV cable. Acetylene black is the industry goal in terms of surface quality and background dispersion. Visually the surface has a matt rather than a shiny finish. Indeed the results confirm the excellent smoothness offered by these formulations. On the other hand the acetylene black has a lower bulk density and this powder is more difficult to wet and then disperse. This manifests itself in higher viscosity and can be more difficult to extrude for the end user.

New developments in furnace grade carbon blacks are all aiming at achieving similar smoothness and performance to acetylene blacks. This means that the black itself must be manufactured from cleaner raw materials and also the carbon black production process must avoid the introduction of extraneous ionic species.

CONCLUSIONS

The production of high quality semiconductive compounds requires a combination of factors. It is usually preceded by a considerable development program in order to establish the formulation.
It brings together along the overall manufacturing chain; raw material sourcing, carbon black quality, compounding and dispersion. The performance of the carbon black is directly related to its physical and chemical properties. Dispersion is also influenced by the carbon black morphology. Loading of carbon black can in itself play a role since the necessary shear mixing can be increased or decreased according to the level of carbon black. Finally it is the compounding step that is responsible for creating the matrix in which the carbon black filler is dispersed and creates the semiconductive properties essential for its end use

application.

The paper presents work conducted on three different carbon blacks all used or intended to be used in semiconductive application. The compounding system was able to clearly differentiate between the carbon blacks and to elaborate the associated parameters of viscosity and electrical properties that accompany the dispersion.

Acknowledgments

REFERENCES

[1]   S. Greene, C.G. Richardson, 1996, "*Dispersion and surface finish, key aspects in compounding of polyolefin based semiconductive products",* Antec, USA.

[2]   G. Matey, C.G. Richardson, 1991, "*Ion migration in aged power cable*", Jicable 3rd International Conference on Insulated Power Cables.

[3]   D.Parfitt, 1991 "*Dispersion of Powders in Liquids*," Applied Science Publishers, London, UK.

[4]   C. Rauwendaal, 1994, *Mixing and Compounding of Polymers,* Hanser, New York, 735-760.

[5]   S. Jakopin, P. Franz, 1983 "*Flow behaviour in Continuous Kneader and its Effect on Mixing*," AICHE Diamond Jubilee, Washington, DC

GLOSSARY

MKS: Modular Kneader System
HV: High Voltage