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Powder Compaction Molding of Bipolar Plates Effect of polymer/graphite composition and processing parameters.

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Gordon 2005, 17 July to 22 July.

I. Introduction.

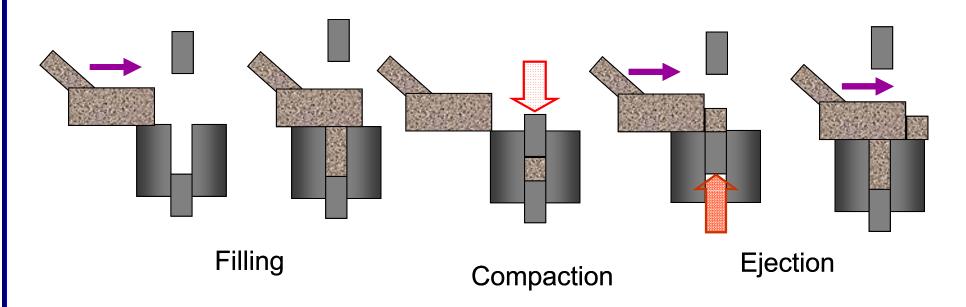
There are different materials used to produce bipolar plates.

- 1. Machined graphite
- 2. Metals
 - Metals/alloys not protected
 - Metals/alloys protected
- 3. C-C composites or molded composites
 - Injection, compression, hot pressing, etc...

Requirements for bipolar plates.

- Good electrical (R < 0.1 Ohm cm) and thermal conductor
- Light and compact
- Gas separator (low H₂ permeability)
- Low cost (< 10\$/kW)
- Mechanically stable (flexural strength > 25 MPa)
- Chemically stable (i.e. corrosion resistant)

A new method of production of bipolar plate based on powder metallurgy techniques was evaluated in this study.



This method enables

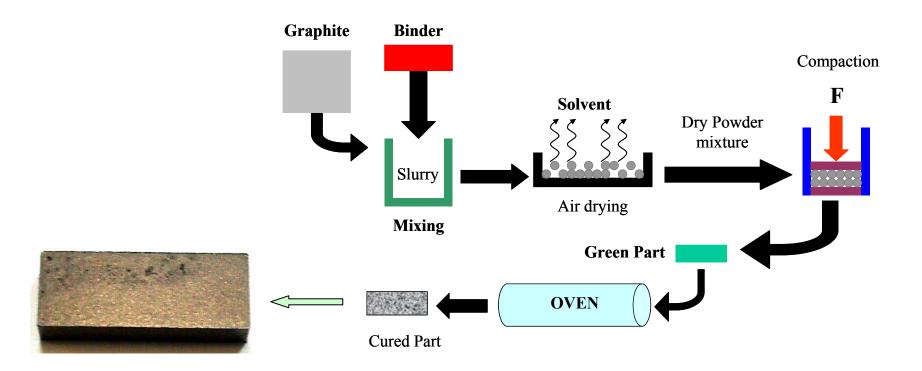
- Mass production (5 to 12 pieces per minute) at low cost
- High graphite loading (higher than 80 wt.%)

II. Experimental.

10 kinds of graphite

- Asbury Carbon: 1645, 3429, 3539, 3610, 4012 and 4956.
- Timcal Graphite & Carbon: SFG-75, SFG-150 and KS-150.
- Conoco: TC-300-L.

Wet mixing with phenolic resin at 5, 10, 15 wt%



- The parts were placed on a glass slide in a quartz tube. A flux of air (500 sccm) was circulated in the tube. The tube was placed in a split-oven and the temperature was increased at a rate of 1 °C/min until 225 °C. After a 20 min hold at 225 °C, the oven temperature was allowed to cool down naturally to 20 °C.
- Rectangular bars (1.248''x 0.497''x 0.25'') were compacted at different pressures, 7, 10, 20, 30 and 40 tsi at a compacting rate of 300 tsi/min. The ultimate pressure was maintained for 3 seconds.
- Cylindrical specimens of 2.54 cm diameter at different heights (0.06, 0.1, 0.2, 0.3, 0.6, 0.9 cm) were also compacted at different pressures, 7, 10 and 20 psi.

The effect of the compacting pressure and the polymer concentration on the following properties was studied:

- Density
- Flexural strength, by Transverse rupture test (TRS) measurement.

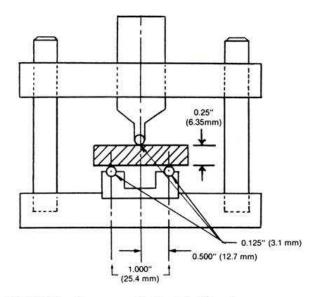
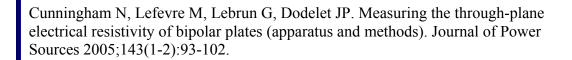


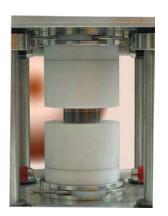
FIGURE 2: Transverse Rupture Test Specimen

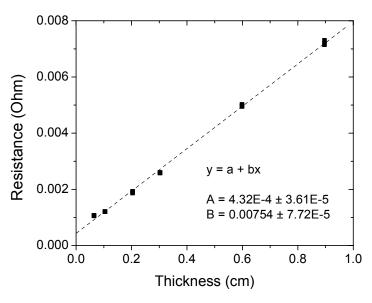
• Resistivity (through plane).

The resistivity of the part was measured at different thickness and the slope of the data multiplied by the surface gives the through plane resistivity.









III. Results.

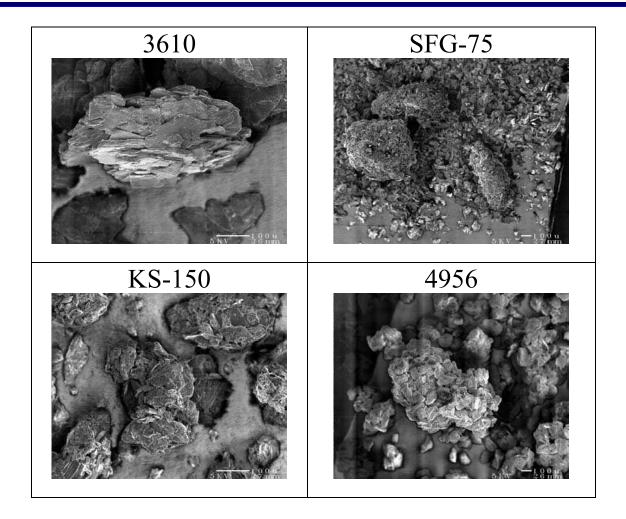
A. Characteristics of graphite powders and graphite-polymer mixtures.

Graphite powders

| I.D. | Туре | D10 (μm) | D50 (μm) | D90 (μm) | Mean particle size (μm) | Apparent Density (g/cm ³) |
|---------|-----------------------|----------|-------------|-------------|-------------------------------|---|
| 3539 | Crystalline flakes | 124 | 204 | 247 | 190 | 0.577 |
| 3429 | Crystalline flakes | 29 | 151 | 236 | 140 | 0.497 |
| 3610 | Crystalline flakes | 34 | 131 | 207 | 130 | 0.392 |
| 1645 | Crystalline flakes | 4 | 15 | 39 | 15 | Nil |
| SFG-150 | Anisotropic synthetic | 21 | 76 | 169 | 70 | 0.263 |
| SFG-75 | Anisotropic synthetic | 10 | 31 | 70 | 36 | 0.236 |
| KS-150 | Synthetic | 15 | 80 | 197 | 80 | 0.344 |
| 4012 | Synthetic | 62 | 116 | 180 | 120 | 0.600 |
| 4956 | Hybrid | 78 | 123 | 167 | 110 | 0.689 |
| TC-300L | Synthetic | 140 | 254 | 452 | 250 | 0.565 |

Graphite-polymer mixtures

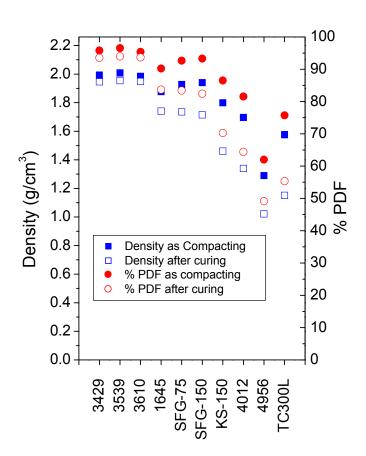
| I.D. | D10 | D50 | D90 | Mean particle | Apparent |
|---------|------|------------|------|---------------|------------------------------|
| 1.D. | (µm) | (µm) | (µm) | size (μm) | density (g/cm ³) |
| 3539 | 172 | 298 | 527 | 324 | 0.498 |
| 3429 | 154 | 297 | 571 | 339 | 0.514 |
| 3610 | 118 | 238 | 462 | 262 | 0.414 |
| 1645 | 11 | 62 | 320 | 128 | 0.430 |
| SFG-150 | 42 | 118 | 228 | 127 | 0.325 |
| SFG-75 | 16 | 47 | 218 | 77 | 0.339 |
| KS-150 | 31 | 126 | 234 | 130 | 0.402 |
| 4012 | 80 | 138 | 216 | 143 | 0.509 |
| 4956 | 98 | 165 | 430 | 213 | 0.590 |
| TC-300L | 173 | 311 | 505 | 325 | 0.498 |

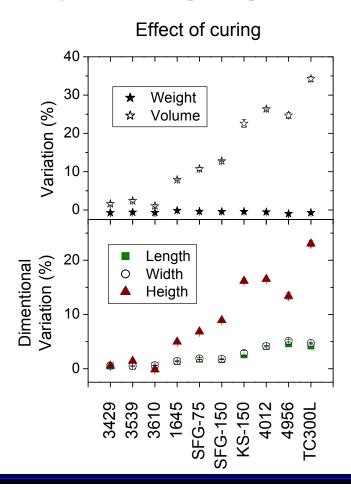


- For 1645 the agglomeration is very effective (15 to 129 μ m).
- For other graphite, the particles size increase around by a factor two.
- The apparent density increases with mixing for graphite with small particle.
- For hard graphite, there is a decrease of the apparent density. There are no very small particles to fill the voids.

B. Effect of the polymer/graphite composition and processing parameters on the density.

- Pure graphite has a density of 2.26 g/cm³ and the density of phenolic resin is 1.2 g/cm³. The theoretical density for a 10 wt. % mixture is therefore 2.15 g/cm³.
- Practically, we don't obtain this density because:
 - o After compaction, there is relaxation of the part which is due to elastic deformation
 - o The different compressibility of various graphite powders.
- When the resin is heated during the curing stage, the resin softens and allows for more relaxation increasing therefore all the dimensions (especially the height) of the compacted part.



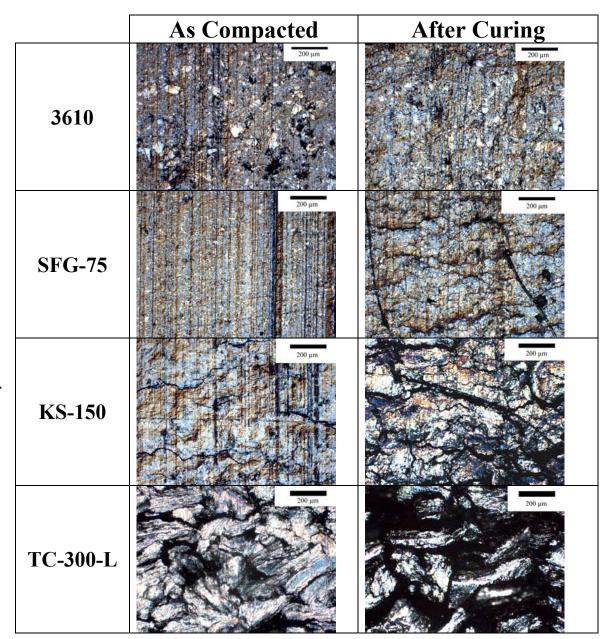


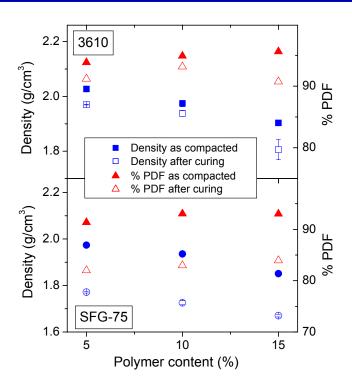
Optical microscopy: side of the parts.

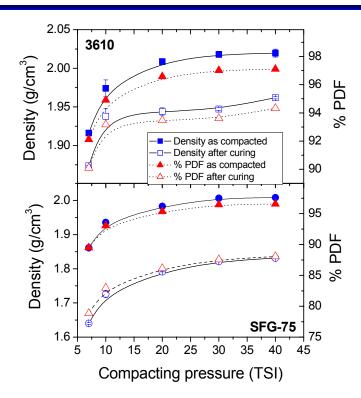
• Only parts made with 3610 family (3610, 3539, 3429) doesn't show delamination.

- For other graphite, cracks are present on the side of the part.
 - Maybe due to the presence of air entrapped in the as compacted parts.
 - And may be due to gases evolved during the curing of the phenolic resin for cured parts.

• With hard graphite porous parts were obtained

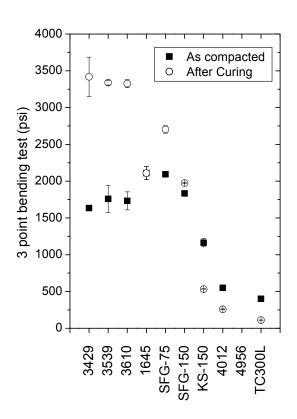


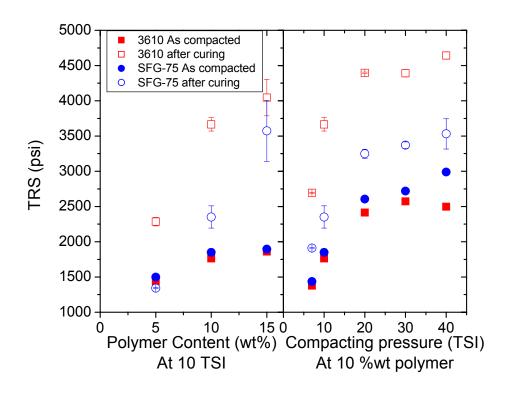




- As expected, an increase of the concentration of polymer implies a diminution of the density of the parts.
- The part, at 15 %wt of 3610, showed visible bubbles. A reason is that the gazes have difficulty escaping the part during curing. Maybe the polymer fills in the low amount of pores and keeps the gases trapped inside the part. Also, there are more gases emitted during curing parts at 15 %wt than at 10%wt.
- The density increase us compaction pressure shows an asymptotic behaviour. For 3610, after 20 TSI, the difference between the dimensions as compacted and after curing has increased due to expansion of entrapped gasses. This effect is not present for SFG-75, since these parts have cracks on the side, allowing the gazes to escape.

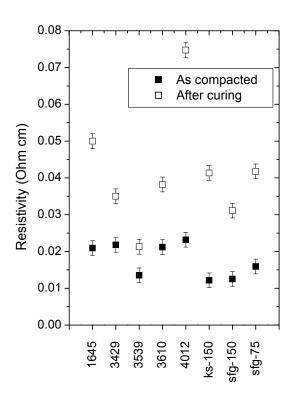
C. Effect on the mechanical properties.

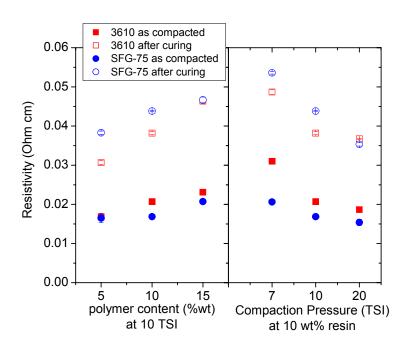




- Except for hard graphite, all as compacted parts have similar TRS.
- Curing increases the TRS for the 3610 family. For SFG and 1645, the increase is small, due to the presence of cracks.
- Increasing the polymer content leads to better mechanical properties.
- Increasing the compaction pressure increases the mechanical properties that reach a plateau after 20 TSI. The increases in density and mechanical properties appear to follows a similar pattern.

D. Effect on the resistivity.





- Before curing, all parts show a similar resistivity around 0.02 Ohm cm. After curing, the resistivity is around 0.04 Ohm cm. Poco graphite has a resistivity of 0.00012 Ohm cm.
- Increasing the polymer loading increases the resistivity.
- Increasing the compaction pressure decreases the resistivity. As apposed to what was observed for the density and mechanical properties, the resistivity increases with higher polymer content and decreases with higher compaction pressures.

IV. Conclusions.

- We were able to produce graphite parts using compaction molding. This low cost technique is applicable to the production of bipolar plate.
- We need a graphite which deforms in a plastic manner for making dense parts.
- The mechanical resistance of the parts is good, around 25 MPa.
- The electrical resistivity of the parts is good, around 0.03 Ohm cm.
- From the results, parts made with 3610 at 10 %wt of graphite and compacted at 20 TSI present a good choice for making bipolar plates.

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