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The analysis of the electrical conductivity of graphite powders during compaction

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Abstract. The conductivity of four graphite powders was measured as a function of the volume fraction of graphite (or air) along (axial) and across (transverse) the direction of compression. The conductivity varies by between two and four orders of magnitude during these measurements. The results are shown to fit an equation which expresses the conductivity of the mixture as a function of the conductivity of the graphite and two morphology parameters. One parameter is the critical volume fraction, at which the graphite–air mixture would become insulating (percolation threshold) and the other an exponent is a combination of the effective demagnetisation coefficients of the grains and the critical volume fraction.

Electron micrographs of the powder grains are shown and volume orientation percentage measurements of the graphite using x-rays were made in order to try to understand the results better.

1. Introduction

The problem of the electrical conduction in heterogeneous systems is an old and difficult one. The ratio of the conductivities of the components can easily be as high as $10^2$ and the conductivity of the medium (mixture) can lie anywhere between the conductivities of the components depending both on the volume fractions of the components and the morphology of the medium. The media can for instance consist of two solids (e.g. cermets), two liquids (emulsions) or a solid and a liquid (suspensions) but this paper will concentrate on the conductivities of graphite powders as a function of the volume fractions of air or graphite, for a powder being compressed between two conducting electrodes. The method of analysing the results can, in principle, be applied to other powders or any binary mixture or medium.

The analysis of such results has previously been made in terms of various phenomenological models, effective medium theories or percolation theory. For instance Marinkovic et al (1971) analysed the resistivities of graphite powders using various phenomenological power laws. One interesting aspect of their results is that there appears to be two linear regions, one at low pressures (below about 6.4 MPa) and the other at higher pressures. The exact pressure where the change-over occurs depends on the packing pressure used in preparing the powder sample. Scarisbrick (1973) analysed his results on carbon-black powders and polymer mixtures as well as colloidal silver films in terms of still other phenomenological models. These two papers contain only some of the phenomenological models introduced, over the years, to describe the conductivity of binary mixtures or compressed powders. The technological importance and uses of such systems are briefly discussed in both Marinkovic et al (1971) and Scarisbrick (1973).

Landauer (1978) traces the theory of the electrical transport in heterogeneous media from the early effective medium theories of Maxwell through the Clausius–Mossotti relation and the Bruggeman symmetric and asymmetric medium theories to the more modern percolation theories (Zallen 1983, Deutscher et al 1983). Coutts (1976) showed that a large number of metal–insulator systems, including those of Scarisbrick (1973), obey the continuum percolation equation

$$\sigma \propto (f' - f_c)^\tau.$$  (1)

Here $f$ and $f_c'$ are the volume fraction and critical volume fraction of the conducting component and $\tau$ is the exponent. $f_c'$ is the volume fraction at which the system or medium becomes insulating. Although percolation theory is only supposed to hold for small $(f' - f_c')$, equation 1 has been observed to hold over...

The purpose of this paper is to show that the conductivity results for graphite powders can be fitted using a new semi-phenomenological effective medium equation developed by McLachlan (1985, 1986a, b, 1987) which holds for media whose morphologies are those of the symmetric and asymmetric media of Bruggeman (Landauer 1978) or lie between these two extremes.

The generalised effective medium equation proposed by McLachlan (1986b, 1987) is

\[
\frac{f(\Sigma_l - \Sigma_m)}{\Sigma_l + [f_c/(1 - f_c)]\Sigma_m + (1 - f)(\Sigma_h - \Sigma_m)} = 0
\]  

(2)

where $f_c$ is the volume of the low-conductivity component and $f_c' = 1 - f_c$ is the critical low-conductivity volume fraction at which the high-conductivity component first forms a continuous percolation path across the medium. $\Sigma_l = \sigma_l^{1/\beta}$, $\Sigma_h = \sigma_h^{1/\beta}$ and $\Sigma_m = \sigma_m^{1/\beta}$ where $\sigma_l$ is the conductivity of the low-conductivity component, $\sigma_h$ is the conductivity of the high-conductivity component and $\sigma_m(f)$ the conductivity of the medium itself. $t$ is an exponent related to the critical volume fraction $f_c$ and the shapes of the grains making up the medium.

In the appropriate limits this expression reduces to Bruggeman's symmetric and asymmetric medium theories.

If $\sigma_l = 0$ and $\sigma_h$ is finite, equation (2) becomes (McLachlan 1987)

\[
\sigma_m/\sigma_h = (1 - f/f_c)^t
\]

or

\[
(1 - f/f_c)^{-t} = (1 - f/f_c)/(1 - L)
\]  

(2a)

or

\[
(1 - f/f_c)^{-m'f_c}
\]

$t = f_c/(1 - L)$ is used for oriented ellipsoids, with $L$ being the effective demagnetisation factor for oriented low-conductivity grains (Sen et al 1981). $t = m_f$ is used for randomly oriented ellipsoidal grains (Meredith and Tobias 1962, Mendelson and Cohen 1982). Equation (2a) has the mathematical form of the percolation conductivity equation.

Using the high-conductivity component volume fractions $f' = 1 - f$ and $f_c' = 1 - f_c$, equation (2) becomes for $\sigma_h = \infty$

\[
\sigma_l/\sigma_m = (1 - f'/f_c')
\]

or

\[
(1 - f'/f_c')^{-t'/L'}
\]  

(2'a)

or

\[
(1 - f'/f_c')^{-m'f_c'}
\]

$\Sigma_l$ and $\Sigma_h$ are the volume fraction at which the high-conductivity oriented grains and $m'$ is used for randomly oriented high-conductivity ellipsoids. Values of $m$ and $m'$ as a function of the demagnetisation coefficient $L_c \neq (L_h = L_l)$, calculated for $\sigma_l = 0$ and $\sigma_h = \infty$ respectively, from equation (23) in Meredith and Tobias (1962), are given in McLachlan (1987).

In the low-$f$ and low-$f'$ limits equations (2a) and (2'a) become independent of $f_c$ and $f_c'$ and depend only on the values of $L$ and/or $L'$, $m$ and $m'$, i.e. on the shape and orientations of the dilute dispersed phase. Values for $L$, $L'$, $m$ and $m'$ can be obtained from equations (2a) and (2'a), which give

\[
L = 1 - f_c/t
\]

(3a)

\[
L' = f_c'/t
\]

(3b)

\[
m = t/f_c
\]

(3c)

\[
m' = t/f_c'
\]

(3d)

It should be noted that for an asymmetric medium, where insulating grains are coated by a conducting component, $f_c = 1$ ($f_c' = 0$) and $L' = 0$ (i.e. the current will always try to remain in the continuous sponge). In an asymmetric medium, where the low-conductivity component coats the very highly conducting grains, $f_c' = 1$ ($f_c = 0$) and $L = 1$ (i.e. the current tries to avoid flowing through the continuous low-conductivity sponge).

Equations (2) and (2a) have been justified by accurately fitting a large amount of experimental conductivity data to them in McLachlan (1986b, 1987). These equations should also apply to the dielectric constant, thermal conductivity and magnetic susceptibility of binary mixtures. In McLachlan and White (1987) it is shown that the magnetic susceptibility data for various binary systems consisting of various amounts of one magnetic and one non-magnetic component could also be fitted using this equation.

The present system is different from those previously analysed, not only in how the volume fraction of each component is varied, but also because the conduction component (graphite) has a highly anisotropic conductivity. The use of these equations in this situation cannot be rigorously justified but they do fit the experimental data very well.

2. Experimental method

2.1. The graphite powders

Four locally available graphite powders were used in these experiments. These are called Natural Flaky Graphite (NFG; Ringsdorff Carbon Corp.), Technical Graphite Powder (TOP; Protea Laboratory Services), Synthetic Graphite Powder (SGP; BDH Chemicals Ltd) and Electrographite Dust (EDG; Ringsdorff Carbon Corp.).

The powders were sized using a Coulter counter. For the NFG about 96% of the particles lie between 20 and 150 $\mu$m. For the TOP about 94% lie between 1.6 and 32 $\mu$m. For the SGP about 95% lie between 1.6 and 32 $\mu$m.
Electrical conductivity of graphite powders

EGD about 90% lie between 106 and 1680 μm. Histograms show that none of the particle size distributions is Gaussian. The NFG and EGD follow a Rosin–Rammel distribution which is

\[ Y = 100 \left(1 - \exp\left[-\left(\frac{x}{k}\right)^n\right]\right) \]

(Rosin and Rammler 1933). Here \( x \) is the particle size, \( k \) and \( n \) are constants and \( Y \) the weight percentage of particles smaller than size \( x \). The parameters obtained are for NFG: \( k = 96 \mu m \) and \( n = 0.90 \) and for EGD: \( k = 770 \mu m \) and \( n = 0.05 \). The other two powders do not obey this nor any other size distribution function. No correlation could be found between the sizes or size distributions and the parameters obtained from fitting the experimental results to equation (2).

Visual examinations were made of all the powders using a stereo microscope for the EGD powders and a scanning electron microscope for all four powders. Before insertion into the electron chamber the powders were sprinkled onto half-dry Aquadag solution which had previously been painted onto a standard aluminium stud. As shapes are difficult to describe, figure 1 shows scanning electron micrographs of all four powders. As can be seen, the EGD particles are the most 'spherical' of all the powders. Both the TGP and SGP powders would appear to consist of a mixture of large flake-like particles and smaller more 'spherical' particles. The TGP flakes appear to be somewhat irregular in appearance while the SGP flakes have a thick layered structure. The SGP grains are probably the second most 'spherical'. The NFG grains are all thin flakes and obviously the least 'spherical'.

Another characterisation measurement made was a determination of the volume percentage orientation of graphite using x-rays. The volume orientation percentage gives the volume percentage of the sample for which the \( c \) axis is parallel to the direction of compression (axial direction). Due to the laminar structure of the grains this is highly correlated with the grain orientation. For the first of these measurements highly compacted pills were made from all the powders in the press which was used for making the conductivity measurements. All the pills except the one made from the EGD remained compacted, without a bonding agent, on being removed from the cylinder. The EGD powder was also the only powder in which the density of graphite (2.26 g cm\(^{-3}\)) could not be reached using a pressure of 300 MPa. The volume percentage orientation in the graphite powders, at or near full compaction, are NFG 96%, TGP 86%, SGP 82% and EGD 30%. A second set of measurements was made on loosely packed powders. The graphite was first poured into a measuring cylinder through a large glass funnel while moving the stream of graphite in a circular motion around the walls of the funnel. From here the graphite was poured in a similar way into an open pan with a removable bottom. After the graphite was sealed in, the pan was inverted and the 'removable' bottom was taken away. This surface of the graphite was x-rayed. Unfortunately as the graphite tended to pack or settle during this procedure the exact volume fraction of graphite is not known, but is probably in the region of 25 to 40%. The percentage orientation measured was NFG 96%, TGP 65%, SGP 19% and EGD 0%.

Figure 1. Scanning electron micrographs of the various powder grains: (a) NFG; (b) TGP; (c) SGP; (d) EGD. A separate scale, to give an idea of the size of the grains, is shown on each micrograph.
The apparatus in which the pills were compressed and which two close-fitting conducting tungsten carbide pistons were inserted. This cell was placed in a hydraulic press. A known weight, which gave a column of graphite about 5 cm in the cylinder, was poured into the apparatus using the method previously described for pouring the loose powders. The top piston was then gently placed on top of the powder and rotated once. The position of the piston, and hence the sample length, was then measured manually and recorded using a linear displacement transducer after which a conductivity measurement was made. After one or two strokes of the pump (one stroke corresponds to a displacement of about 0.4 mm) the new position of the piston was recorded using the transducer and the conductivity was measured again. This procedure was repeated until the maximum allowable pressure was reached. Here another manual measurement of the piston position, to complete the calibration of the transducer, was made. During the last few readings the pressure in the hydraulic system was high enough to be read on the gauge provided. The pressure during these measurements went from 20 kPa (due to the weight of the piston) to 300 MPa.

Transverse measurements were made in a square cell. The cylinder consisted of two copper plates separated by two Perspex side walls, a short square Perspex plunger at the bottom and a long one at the top completed the cell. The same loading and measurement procedures were used. In this case the pressure went from 1 kPa to 50 MPa.

Low conductivities were measured by applying a constant 100 mV across the cell and recording the resulting current. When the conductivity became too high the voltage dropped below 100 mV and both voltage and current readings were taken. All the measurements were made using a computer-controlled data logger system. The conductivities (from the current and voltage) and the length of sample (from the displacement transducer) were calculated in this system. The results were then computer fitted to equation (2).

### Table 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\delta$</th>
<th>$f_1$</th>
<th>$t$</th>
<th>$\sigma$ ($\Omega^{-1} \text{m}^{-1}$)</th>
<th>$m$</th>
<th>$m'$</th>
<th>$L$</th>
<th>$L'$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NFG</td>
<td>0.5</td>
<td>0.295 ± 0.004</td>
<td>1.94 ± 0.05</td>
<td>9000 ± 900</td>
<td>2.76</td>
<td>6.80</td>
<td>0.64</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>0.4</td>
<td>0.27 ± 0.02</td>
<td>2.8 ± 0.3</td>
<td>113000 ± 11000</td>
<td>3.80</td>
<td>10.20</td>
<td>0.74</td>
<td>0.10</td>
</tr>
<tr>
<td>T</td>
<td>0.3</td>
<td>0.315 ± 0.01</td>
<td>2.71 ± 0.05</td>
<td>1160 ± 100</td>
<td>3.96</td>
<td>8.61</td>
<td>0.75</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>0.4</td>
<td>0.33 ± 0.01</td>
<td>2.7 ± 0.1</td>
<td>45000 ± 10000</td>
<td>4.03</td>
<td>8.18</td>
<td>0.75</td>
<td>0.12</td>
</tr>
<tr>
<td>T</td>
<td>0.6</td>
<td>0.27 ± 0.01</td>
<td>1.52 ± 0.03</td>
<td>14700 ± 1500</td>
<td>2.08</td>
<td>5.63</td>
<td>0.52</td>
<td>0.18</td>
</tr>
<tr>
<td>SGP</td>
<td>0.7</td>
<td>0.25 ± 0.02</td>
<td>2.1 ± 0.2</td>
<td>50800 ± 15500</td>
<td>2.80</td>
<td>8.40</td>
<td>0.64</td>
<td>0.12</td>
</tr>
<tr>
<td>EGD</td>
<td>0.5</td>
<td>0.38 ± 0.04</td>
<td>1.5 ± 0.4</td>
<td>79000 ± 25000</td>
<td>2.48</td>
<td>4.05</td>
<td>0.60</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>0.5</td>
<td>0.37 ± 0.015</td>
<td>2.4 ± 0.5</td>
<td>140000 ± 22000</td>
<td>3.80</td>
<td>6.58</td>
<td>0.74</td>
<td>0.15</td>
</tr>
</tbody>
</table>

#### 2.3. Computer fitting procedure

Either equations (2) or (2a) could be used to fit the data. In practice equation (2) was used with the conductivity of air being fixed at $10^{-9} \text{\Omega}^{-1} \text{m}^{-1}$ i.e. at a value 1000 or more times smaller than the maximum value observed for compacted graphite. The variable parameters used in fitting the data are the conductivity of the graphite, the critical volume fraction $f_c$, the exponent $t$ and the length of the sample $l_c$ when the graphite is 100% compacted. $l_c$ is needed to calculate the correct volume fraction of air from the variable length of the sample $l$ using the expression $(1 - l/l_c)$. As $l_c$ is either accurately known or can be closely estimated from the compaction of the powder or the density of graphite, it is only allowed to vary between narrowly controlled limits.

The program uses the experimental conductivity and the variable parameters to calculate a volume fraction called Fit from equation (2). Fit is then compared to the volume fraction (Fm) calculated from the sample length $l$ and $l_c$ and the quantity

$$\chi^2 = \sum_n [(\text{Fit} - \text{Fm})/0.01]^2$$

is minimised by the program by varying the non-fixed parameters. If $\delta = (\chi^2/(N - P))^{1/2} = 1$, where $N$ is the number of experimental points and $P$ the number of variable parameters, it is claimed that the data have been fitted to an accuracy of 0.01 in the volume fraction. $\delta$ and the errors given with the fitted parameters are calculated by the program.

#### 3. Results and discussion

The results of fitting the conductivity data to equation (2) or (2a) are given in table 1. In the first column is the sample name and the A and T in the second column stand for axial and transverse measurements respectively. $\delta$, as defined above, is the average $\delta$ for all this particular type of experimental run. Similarly $f_c$, $t$ and $\sigma$ are also average values for a set of measurements, e.g. NFG type A. The errors are the standard deviation of results for a particular set of measurements. At least
Figure 2. The log–log plots of the conductivity of the NFG as a function of the volume fraction of graphite. The axial experimental points are marked + and the transverse ones x. The full lines are the best fits to equation (2). For these particular runs the axial parameters are \( \delta = 0.34, f' = 0.299 \pm 0.04, t = 1.92 \pm 0.03, \sigma = 8670 \pm 90 \, \Omega^{-1} \, \text{m}^{-1} \); and the transverse parameters are \( \delta = 0.19, f' = 0.267 \pm 0.002, t = 2.69 \pm 0.02, \sigma = 115700 \pm 1000 \, \Omega^{-1} \, \text{m}^{-1} \).

Figure 3. The log–log plots of the conductivity of EGD as a function of the volume fraction of graphite. The axial experimental points are marked * and the transverse ones x. The full lines are the best fits to equation (2). For these particular runs the axial parameters are \( \delta = 0.40, f' = 0.396 \pm 0.005, t = 1.21 \pm 0.04, \sigma = 75900 \pm 2400 \, \Omega^{-1} \, \text{m}^{-1} \); and the transverse parameters are \( \delta = 0.22, f' = 0.345 \pm 0.003, t = 2.75 \pm 0.07, \sigma = 139300 \pm 6700 \, \Omega^{-1} \, \text{m}^{-1} \).

two, but usually three or four, sets of measurements are made in each case. Some of the results for the NFG (least 'spherical') and EGD (most 'spherical') are shown in figures 2 and 3. Similar plots are obtained for the SGP and TGP powders.

For a single set of measurements on a particular powder the errors for the variable parameters \( f', t \) and \( \sigma \) are considerably lower than those given in table 1 as shown by the small average values of \( \delta \), for all samples, given in column three and the data shown in figures 2 and 3. One possible cause of this for this variation in the fitted parameters could be that samples with different size distributions were poured from the container for the various measurements made on the same powder.

From figures 2 and 3 it can be seen that the data have not been fitted to highest volume fraction of graphite measured, nearly 1.0 for NFG, TGP and SGP in axial measurements at the highest pressures. If the data are fitted using these points, the \( \delta \) obtained are about three times larger and the highest graphite volume fraction points obviously give the largest \( \chi^2 \) values. Upon examination the large contributions to \( \chi^2 \) were found to occur where the pressure on the sample exceeded 50 MPa. This is thought to mark the onset of compaction by crushing as well as slippage and so no data where the pressure exceeded 50 MPa were included in the analysis. This also eliminates serious pressure effects on the conductivity of the graphite itself. This shows that, not surprisingly, a single pair of morphology parameters can only be obtained for a particular type of compaction or agglomeration process.

The difference between these results and the two linear region ones of Marinković et al (1971) is primarily due to the method by which the samples are prepared and their geometries. The present experiment starts in
an 'as-poured' state (length = 5 cm), and the final sample is about 2 cm (diameter 2.6 cm). Marinkovic and co-workers samples are prepared by pouring powder into their cylinder (diameter 1.1 cm) and tamping it down to a length of about 1.5 cm at the packing pressure for that specimen. This process was repeated about 10 times to create a 'multi-layer' specimen. The experimental results which are shown in their paper are for specimens with a very different initial state from those described in this paper.

From table 1 it can be seen that the NFG and SGP powders have the smallest critical volume fractions of graphite \( f_c \) at the 'metal–insulator' transition. These two powders have the most regular layer-like structure. The approximate orientation of the graphite at the first measured point has been previously given. It may be assumed that at the 'metal–insulator' transition the NFG graphite flakes are oriented and that the SGP and EGD powders are fairly or completely randomly packed. The orientation for the TGP powder at \( f = f_c \) is not clear, but it should be noted that the TGP powder is the only insulating one in the 'as-poured' state and that the volume fraction of graphite at this point was lower than the \( f_c \) determined from equation (2). Unfortunately no real conclusions can be made about what actually determines the critical volume fraction. Remarks concerning the values of \( r \) are incorporated in a later discussion on the effective demagnetisation coefficients \((L, L', m, m')\) which are calculated from equations (3), and listed in table 1, as are the mean fitted values of the axial and transverse conductivities of the different graphites.

One of the most notable features of the values of \( \sigma \) listed are the low values obtained, in both the axial and transverse case, for TGP. This must be assumed to be due to the chemical composition or, possibly, the microscopic structure of the TGP graphite grains and no further comment will be made. As might be expected, the largest ratio of the axial and transverse conductivities is for NFG, which is the most oriented (96%) and which has the most flaky structure of the powders. Conversely, the smallest ratio is for the least oriented, most 'spherical' EGD. The ratios for TGP and SGP are very similar, as are the percentage orientations near full compaction. Therefore it would appear that the percentage orientation, as measured by x-rays, is highly correlated with the axial-transverse conductivity ratio.

As stated in the Introduction, a high value of the effective demagnetisation coefficient \( L \) from equation (3a)) for the insulating component (air) corresponds to this component wrapping around or nearly completely coating the conducting particles (graphite grains) in the high volume fraction of graphite regime. This explanation of the reasonably high values of \( L \) would appear to be in agreement with the morphology of compressed graphite powders.

As no measurements are (or can be) done in the dilute graphite region beyond the 'metal–insulator' composition a lengthy interpretation of the \( L' \) (equation (3b)) or \( m' \) (equation (3d)) coefficients is not made. Low \( L' \)-values for NFG and possibly the TGP powders in the transverse direction is consistent with current flow in the plane of the oriented graphite flakes but the other low values of \( L' \), especially for NFG type A, are then inexplicable. The values of \( m' \) are all consistent with what is to be expected for randomly oriented platelets (see figure 1 in McLachlan 1987) and this is probably the best explanation for the TGP, SGP and EGD powders. It is, however, not possible to come up with reasonable models to account for all the values of \( L' \) and \( m' \) obtained. A possible explanation for this could lie in the fact that the conductivity of the powders as well as their shape is anisotropic. This is the first occasion that equation (2) has been used to analyse such a system.

Although equation (2) fits the data very well, especially for a single compaction, the morphology parameters \( \{f, t\} \) are almost certainly dependent on the precise experimental conditions. Rajab and Coleman (1985) show that the density distributions in iron powders, compacted in a piston cylinder apparatus, depend on the type of powder, the length-to-diameter ratio and the density or state of compaction. Our experiments show that powder type has a major effect on the morphology parameters (table 1) and it may be surmised that the final length-to-diameter ratio probably has a minor effect on the morphology parameters. As graphite is self-lubricating and the cylinder walls highly polished, distortion due to the effects of the powder 'sticking' to the walls is probably very small (Thompson, 1981). Also as the cell is very large \((D = 26 \text{ mm}, H(\text{final}) = 20 \text{ mm})\) compared with the powder grain size \((1.6-150 \mu \text{m})\), surface-to-volume ratio effects probably have negligible effect on the morphology parameters. Theoretical treatments of this problem have also been made (Thompson 1981). The variations in the parameters for the same powder may be due to some of the above effects as well as the near impossibility of exactly reproducing the 'as-poured' powder for each experimental run. Also the morphology parameters obtained for the transverse measurements are probably influenced by the fact that a parallelepiped cell has been used. As the pressures used are not excessive and distortions of the apparatus very small compared with the final dimensions of the sample, the cell walls can be assumed to be rigid.

4. Conclusions

Equation (2) can fit the conductivity, as a function of volume fraction, data for very differently shaped anisotropic grains, over several orders of magnitude of conductivity, using only two morphology parameters. The volume fraction was changed by compressing the powder and the morphology parameters obtained appear to change somewhat as the compaction process changes, i.e. from pure slippage to slipping and crushing.

The strong correlation between the x-ray orientation...
results and the ratio of the axial and transverse conductivities is also of interest, as is the way that the graphite orientates during pouring and compaction.

Acknowledgments

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