End-capping and other defects in pressed ceramic compacts

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End-capping defect is one of the causes of rejection in pressed and sintered ceramic compacts. The causes of its occurrence lie both in the quality of the powder and that of pressing. In this paper, end-capping and other defects have been described and their relationship to powder characteristics and pressing parameters has been brought out. It is found that soft powders that are free from agglomerates, and well finished suitably tapered hard material dies and moderate compaction pressures tend to minimise end-capping and other defects.

Pressed and sintered ceramic compacts sometimes exhibit a defect called end-capping (Fig. 1). End-capping was found to occur either separately or in association with other defects, mainly, cracks. Some aspects of these defects were discussed earlier1,2. End-capping is defined as that problem whereby cracks, starting in the upper corner of a compact or pellet, move towards its centre at an angle of 10-20° relative to the top surface3. If the problem becomes severe enough, the top may break out of the pellet, resulting in a circular end-cap of conical shape with a 140-160° included angle.

Even though the defect develops in green stage, it is difficult to detect and usually revealed only on sintering and finish grinding. The simple acetone dip test may be used to detect location of defects at green stage. The size of bubbles evolved from a green pellet dipped in acetone gives an indication of the size of the defect. Sophisticated acoustic emission methods to evaluate green pellets are also developed4.

Stresses in a Compact

Loose powder mass filled in a die undergoes irrecoverable or plastic deformation as the punches compress it from top and bottom. As the applied axial pressure is increased, pressure also gets transmitted radially to the die wall through the powder mass and the forces of reaction from the die wall begin to compress the compact radially. In contrast to metal powder compacts, cold welding is unlikely in ceramic powders which lack ductility. Consolidation takes place by fracture and rearrangement of the fragments. After a certain compactness is achieved, as the applied pressure is increased further, there is some recoverable or elastic deformation in the compact. The compaction die also expands elastically to a small extent depending on die rigidity. Defects in the green compact can originate during the elastic recovery processes of the compact as well as of the die taking place on withdrawal of applied pressure. It has been stated that defects can occur even when very rigid dies are used5.

Consider the stresses in a compact (Fig. 2a). The vertical compressive stresses are maximum at the punch faces and decrease on moving towards the centre of the compact. The radial stresses are maximum at the die wall and decrease on moving towards the centre. The resultant stresses are maximum at the corners as shown (Fig. 2b). Stress

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relaxation is, therefore, likely to be maximum at
the corners in opposite directions.

On removal of the punches after compaction is
over and while the compact is still in the die, the
compact experiences radial compression by die
wall and tends to expand axially by Poisson effect.
When the axial expansion stress exceeds the green
strength, fracture can occur while the compact is
still in the die. The radial expansion stress within
the compact is balanced by reaction from the die
wall as long as the compact is contained in the
die (Fig.3a). On ejection from the die, the com­
pact tends to expand radially since die wall rea­
tion is no longer available to oppose expansion
(Fig.3b). When the radial expansion stress exceeds
the green strength, fracture again occurs as the
compact leaves the die. Circumferential lamin­
atios appear on the sintered compact on finish
grinding.

Consider a perfectly elastic material under
compaction pressure in a frictionless die (Fig.4a).
On withdrawal of the punches, the elastic material
expands axially and uniformly across the diameter
as the elastically expanded die tends to return to
its original dimension. This is the case when there
is no friction between die wall and compact. If the
material is non-elastic, fractures is possible.
Where die wall friction exists, at the edges of the
compact, the axial stress is balanced by it and
there can be no expansion at the edges (Fig.4b).
Under such a condition, axial stress relief in the
compact on punch withdrawal is not complete.
The magnitude of the unrelieved axial stress in­
creases as the die wall-compact friction increases.
This residual axial stress gets relieved on ejection
of the compact from the die.

A compact leaving the die, therefore, comes un­
der the influence of two types of stresses. One is
the radial expansion stress and the other is axial
residual stress. As the compact leaves the die with
wall friction, stress relief occurs both axially and
radially. When the resultant stress exceeds the
green strength, end-capping defect can occur in
the direction of the resultant stress. The axial re­
sidual stress is maximum at the commencement
of ejection and its relief is complete when ejection is
complete. Hence end-capping is most likely to oc­
cur at the instant when the first end of the com­
pact begins to leave the straight portion of the
die. It is top end-cap. The angle of end-capping
depends on the relative magnitudes of the axial

Fig. 2—Compaction stresses in a section of a compact in a
double acting press (a) axial and radial stresses, (b) resultant
stresses

Fig. 3(a) Compact within a die on punch withdrawal with ra­
dial stress balancing die reaction. (b) Ejected portion after
stress relief and radial expansion

Fig. 4—Axial expansion within die of a perfectly elastic com­
pact on punch withdrawal (a) die wall friction zero, (b) die
wall friction significant
and radial residual stress reliefs. End-capping can occur only when there is residual axial stress in the compacts which, in turn, is caused by die wall friction. The friction force depends on the coefficient of friction between the compact and the die wall (which in turn depends on powder hardness, die barrel finish and lubrication) and the compression from die wall (which in turn depends on powder hardness, die rigidity and compaction pressure).

When a press feed shoe moves over die holes, fine particles in the powder are the last to fall into die cavity forming a layer over coarser granules. These fines being nearest to the punch, experience high compaction pressure. However, the fines cause a low pressure area immediately below them in the compact. The pressure loss is due to larger interparticles friction between fine particles. Such a situation is conducive for end-cap formation and even separation of the end-cap from the compact.

**Experimental Procedure**

Uranium dioxide powder of specific surface area 3 m²/g (by BET method) and average particles size 2 μm (by Fisher's method) was precompacted at 100 MPa into slugs. The slugs were broken and debris were sieved to -14 and +60 mesh size to form granules. These granules were then compacted at 100-300 MPa into cylindrical shapes of diameter and length of 18 mm to a green density of about 50% of the theoretical density (TD), 10.96 g/cc. For a compaction pressure of 290 MPa, the pressure was brought down to either 62 or 145 MPa in one or four seconds. Diametral expansion of the green compacts after full ejection were noted for different compaction pressures. The compacts were sintered at 1700°C in pusher type sintering furnace in cracked ammonia atmosphere to densities above 95% TD.

**Results and Discussion**

**Detensioning effects**—The applied compaction pressure is brought down to the value of hold down pressure over a period of time before ejection is commenced. The process is called detensioning, the duration of which is found to affect defect formation in powders of low recovery. As the duration is increased, the stress relief defects (end-caps and cracks) decrease (Table 1). On the other hand, the detensioning duration was found to be immaterial in powders of high recovery.

**Die material effects**—Cold die steel expands more than does tungsten carbide, the effects of which are given in Fig.5. For the same compaction pressure the compact expansion is found to be greater when CDS die is used.

**Other effects**—It is found that powders which resulted in end-capping contained hard agglomerates. Deagglomeration may be achieved by milling the powder before compaction. Soft powders lead to higher packing efficiency in compaction and to higher green strength.

As the compaction pressure is increased, a point of diminishing returns is reached in terms of green density and green strength, while the maximum ejection stress increases rapidly, causing cracking. The breakaway ejection force is the force needed to overcome static friction and to expand the die elastically to allow compact movement. Once the compact is moving, the force needed to continue motion decreases, largely due to the difference between static and kinetic friction. The die exit geometry interacts with the expanding compact and controls the stress state associated with part emergence.

It was earlier found that excess lubricant in the bottom punch wets the lower portion of the powder column. The wet portion gets compacted to a greater degree than the dry portion, with a good chance of separating at the dry/wet interface. Further, the deeper the compaction zone in the die barrel, the longer the compact travel during ejection and the more the defects. Excessive hold

<table>
<thead>
<tr>
<th>Compaction pressure, MPa</th>
<th>Pressure on detension, MPa</th>
<th>Detension duration, s</th>
<th>Accepted per cent</th>
<th>Rejected per cent end-caps, cracks</th>
<th>Rejected per cent pits, chips</th>
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</thead>
<tbody>
<tr>
<td>290</td>
<td>62</td>
<td>1</td>
<td>47</td>
<td>53</td>
<td>0</td>
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<tr>
<td></td>
<td>145</td>
<td>1</td>
<td>66</td>
<td>21</td>
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</tbody>
</table>

### Table 1—Effect of detension time on sintered compact acceptance
down pressure on the compact can cause crushing on ejection. The end-capping tendency was more for complex compact geometries. For example, end-caps were negligible in flat faced cylindrical compacts. Chamfered compacts end-capped more than dished compacts. High rates of compaction and defective punch tips were also found to cause end-capping.

Remedial measures—Correct die-punch alignment, secure positioning of punch and die to remain undisturbed through, many cycles of operation, frequent cleaning of granulator sieves, press tooling as well as lubricant channels, sufficiently long residence time of powder feed shoe over die holes (4s) and compaction pressure dwell time (8 s) are prerequisites for defect-free compaction. The dwell time is required for overcoming the inertia of the powder to compact into highest packing efficiency.

The use of low or moderate compaction pressure in highly polished dies, maintenance of a suitable hold-down pressure during ejection and provision of a small taper for facilitating compact ejection are known preventive measures against end-capping in high speed pressing. The taper should always be less than the diametral expansion of the compact. Powder admixed lubrication tends to increase end-capping while die wall lubrication decreases it. Decreasing L/D, increasing hold down pressure and increasing green tensile strength help in minimizing the defect.

An earlier work brought out the effects of die finish, die taper, excessive applied pressure, deep die pressing and speed of ejection. Prevention of collection of excess lubricant on the bottom punch helps minimize bottom end-capping.

The present experimental work has brought out the importance of rigidity of die material and provision of detensioning time. The percentage of endcaps is generally less when tungsten carbide die is used by virtue of its greater rigidity and ability to get fine polished. However, since the diametral expansion of compact is less in rigid tungsten carbide die, the taper also has to be less than in cold die steel die, to minimise end-capping from the more expensive tungsten carbide die.

Elimination of fines from granulated powder before final compaction is another remedial measure. For example, − 14 + 60 mesh granules have given much less end-capping than did − 14 mesh granules.

It should be mentioned here that under the same pressing conditions, some powders result in end-capped compacts while some others do not. Where it is not feasible to take remedial measures such as decreasing die taper or decreasing compaction pressure or increasing hold down pressure, addition of 0.3 weight per cent of zinc benenate powder, Zn(C₂₃H₄₃COO)₂ to the ceramic granules significantly reduces end-capping.

Conclusion
Based on an understanding of the nature of the stresses within a pressed ceramic powder compact in a double acting press, a mechanism for end-capping and related defects has been suggested. In addition to various other factors, die material and post-compaction detensioning time have been found to affect defect formation.

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References