Characterization of Pressing Process of RDX Crystal Grain by Cone-beam Micro-focus Computed Tomography

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Abstract: In this study RDX granular-crystalline in pressing by the cone-beam micro-focus Computed Tomography (µCT) is presented. The granular displacement and inter-granular void are analyzed including crystalline crack and the density distribution. The results not only show that the packing of the RDX crystals becomes more compact and uniform under the pressure with 150MPa, but also manifest some visible micro-voids inside RDX crystals. As a non-destructive technique, it is proved to be high efficiency and strong capability to assess crystalline motion and fracture characterization in pressing by cone-beam µCT. The analysis results indicate the tendency of the displacement of RDX crystals is in nonlinearity, and the crystalline fracture is strongly influenced by the pressure and restricted by shape of the crystal mould. It is also analyzed that the density distribution gradient is obvious along the pressing direction.

Keywords: RDX crystal, pressing process, cone-beam micro-focus computed tomography

1. Introduction

The pressing and molding method is one common-used technique in explosive charging, which is generally processed by pressing the explosive crystalline or polymer bonded crystalline in modest high temperature \[^1\]. Several parameters such as the pressure, crystalline packing style, void fraction and compressibility of sculpting power as well as the shape of mold, height/radius ratio and temperature can influence the microstructure’s stress distribution including the quality and performance of the molded explosive. After decades of researching works, the relationships between explosive quality and its safety, detonation performance and mechanical properties had been understood very well. However there is no similar researching tool or with a slightly better resolution compared to destructive method, nor has the capability to study the internal damage, crack, non-uniformity and some other quality flaws as well as their influence to the performance structure by nondestructively and quantitatively method. All these issues still remain superficial, so how to avoid and control these quality flaws has always been considered as one of the most important tasks in explosive molding technique.

High resolution industrial Computerized Tomography shows strong capability and many advantages in revealing internal structure from lots of researches \[^2-5\]. Especially in recent years, using the micro-/nano-CT to characterize material properties became one of the most concerned topics in material science \[^6-9\]. Our previous study also verified the strong capability of high resolution industrial CT on characterizing defects of explosive single crystal and thermal solidified RDX explosives both qualitatively and quantitatively \[^10,11\]. Hence, comparing to some other methods such as SEM, optical microscope that can only obtain the surface information \[^12,13\], while high resolution industrial CT does have strong and distinct superiority \[^14\]. It provides a useful method for studying the damage, crack and non-uniformity in pressing molding explosives in microscale.
The cone-beam µCT was applied to study the pressing process of RDX crystals in this study. Displacement, void, crack and density distribution during pressing was analyzed as well. The goal of this study is to characterize the pressing process in multiscale (macro/ meso/ micro), which is also important for the study of molding process and its relation to explosive performance.

2. Experimental Setup

2.1 Explosive samples

The explosive used in this experiment is RDX crystal, with theoretical maximum density of 1.816 g/cm$^3$, and the range of size distribution is from 800µm to 1000µm. The inner pores are negligible.

2.2 Experimental Setup

The cone-beam µCT is a third-generation system with a fixed flat source-detector arrangement, and with a translation/rotational movement sample deck. The X-ray source is a 225 kV Fein-Focus spot, which allows for a resolution down to 5 µm for an object of 5 mm. The sample deck rotates and moves automatically along the zoom control axis. The scheme of the CT experimental set-up is illustrated in Fig.1.

Fig.1 The schematics of the µCT experimental set-up.

The mold is made of PEEK, with density (1.665±0.002)g/cm$^3$, tensile pressure more than 200MPa. The size of mold is about Φ50mm×34mm, with a charging part of diameter 6mm. The procedures of experiment are illustrated in Fig.2. First fill the mold charging part with RDX crystals, then make the initial CT scan, and press the crystals with 5MPa pressure(at room temperature, load rate 0.1mm/min) in an material testing machine, unload it and make the second CT scan, then repeat this procedure under different pressure subsequently: 20Mpa, 40MPa, 50MPa, 150MPa. The scan parameters remain the same for each scan, which the voltage and current of the tube are 120 kV and 150 µA and the magnification factor is 20. The voxel size is about 20µm.

3. Results and Discussion

3.1 CT Scan Results
Fig. 3 and Fig. 4 both illustrate some typical slices of CT scan volumes under different pressure. Much phenomena during pressing could be observed from the figures, such as translation of grains, grain cracking, increasing density and filling of inter-grainular space for examples, 1) The initial state of grains was loosely packed, with few contacting to neighbor grains (Fig.3a and Fig.4a); 2) Under pressure with 5MPa, grains on the top part began to crack and small pieces fell into the gap in the bottom part (Fig.3b and Fig.4b). Grains of lower parts weren't affected very much; 3) when the pressure was increasing to 20MPa and 40MPa, the bulk volume had an apparently decrease. The crystal fracture became evident and propagated to the lower part. Through cracks could have big crystals changing into smaller ones. The displacement of crystals was mainly along the pressing direction. Big crystals are contacted to small crystals, more closely and the compactivity increased gradually; 4) when the pressure was higher than 50MPa, the change in displacement, compactivity and filling effect between crystals became less evident (Fig.3e and Fig.4e); 5) when the pressure was increased to 150MPa, horizontal cracks emerged both in the top and bottom parts of the cylinder (Fig.3f).

3.2 Discussion and Analysis

3.2.1 Displacement
Fig. 5a and 5b both give the relative displacement versus the pressure of three typical markers from Fig. 3, the markers remain their characteristics during the pressing process. The results show that under the confinement of the mold, marker crystals moved mainly along the pressing direction, with little radial displacement. The displacement under lower pressure is more evident than the higher pressure. The displacement close to the end of pressing is much more evident than that of the fixed end. When the pressure was increased, the displacement became less evident and varied non-linearly with respect to the pressure (Fig. 5c). This verified the capability of cone-beam uCT to observe the crystal movement non-destructively during the explosive molding process. The CT scan analysis revealed that the molding pressure and the surface of pressing are the main factors that influence on the crystal displacement at different positions.

3.2.2 Crystal void

The filling effect during explosive pressing (or equivalently the void fraction) determines the structure of explosives, and thus the quality of molding. Hence obtaining the void fraction has special meaning for the explosive press molding technique. In this paper, the void fraction (denoted by \( h_i \)) is computed by accumulated the void area of every CT slice:

\[
h_i = \frac{S_i - S_{i,\text{ROI}}}{S_i}
\]

Then the void fraction of entire cylinder can be written as:

\[
H = \frac{1}{n} \sum_{i=1}^{n} h_i
\]

Where the \( j \) is the slice index, \( n \) is the number of slices.

According to formula (1), Fig. 6 gives the void fraction versus slice indices (0 is fixed end, maximum is the pressing end) under different pressure. Table 1 gives the void fraction of entire cylinder (denoted by \( H \)) and the "local" void fraction (at top, middle and bottom 50 slices, denoted by \( H_{1-50} \), \( H_{\text{middle}} \), \( H_{\text{upper-50}} \) respectively). The result shows that the void fraction was considerable by large fluctuation at initial state. When the pressure was increased, the movement, rearrangement or even fracture of crystals effectively filled the space between crystals, which led to an obvious drop of void fraction. Moreover, the fluctuation was also decreased, making the entire cylinder more compact and uniform. Nevertheless, there is an evident decrease with respect to the slice number (i.e., along the pressing direction). When
it is close to the pressing end, the void fraction is less, and that means a more severe fracture of crystals and more complete gap filling. It is evident that the stress of pressing end is more than that of fixed end, and that is probably due to the friction between neighbor crystals and the mold wall.

Table.1 Void fraction at different pressure

<table>
<thead>
<tr>
<th>Load Stress/ MPa</th>
<th>0</th>
<th>5</th>
<th>20</th>
<th>40</th>
<th>50</th>
<th>150</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H$</td>
<td>0.403</td>
<td>0.286</td>
<td>0.237</td>
<td>0.155</td>
<td>0.085</td>
<td>0.027</td>
</tr>
<tr>
<td>$H_{1-50}$</td>
<td>0.417</td>
<td>0.378</td>
<td>0.299</td>
<td>0.216</td>
<td>0.141</td>
<td>0.069</td>
</tr>
<tr>
<td>$H_{middle\pm25}$</td>
<td>0.400</td>
<td>0.283</td>
<td>0.225</td>
<td>0.150</td>
<td>0.076</td>
<td>0.021</td>
</tr>
<tr>
<td>$H_{upper\pm50}$</td>
<td>0.391</td>
<td>0.219</td>
<td>0.204</td>
<td>0.116</td>
<td>0.058</td>
<td>0.007</td>
</tr>
</tbody>
</table>

3.2.3 Cracks

The fracture of crystal is a key factor in explosive molding technique, for it would directly determine the molding structure. Since RDX could be considered as brittle material, the change in crystal volume during press would be ignored. Moreover, it's maybe impossible for RDX crystals to fill the gap without being severely deformed or fractured. Hence, the reduction of void volume (i.e., the increase of "filled" volume) could characterize the degree of crystal fracture (denoted by $K_i$):

$$K_i = \frac{(V_0 - V_{\text{RDX}})/V_0 - (V_{\text{press}} - V_{\text{RDX}})/V_0\times100}{(V_0 - V_{\text{RDX}})/V_0\times100} = \frac{H_0 - H_i}{H_0}\times100\%$$

where $H_0$ and $H_i$ is the void fraction of initial state and under specified pressure, respectively.

According to the formula (3), Fig.7 illustrates the degree of fracture versus pressure. From Fig.7, we can see that large crystals fracture and filling mostly happened at the relative low pressure, while at the medium pressure, the small crystals fractured again but had less void filled, and the fracture at high pressure almost had no obviously change since it is hard for tiny crystals to fracture again. Fig.8 illustrates the fracture of large crystals at 5MPa compared with the initial state.

From Fig.3, Fig.4 and Fig.8, a few holes inside the crystals could be recognized. As observed from the CT volume, the crack propagation usually started from the holes. Some other cracks also formed under contact forces between neighbor crystals. The fracture usually began at the boundary of large crystals, and then the crack propagated until it reached another
end of the crystal. The direction of cracks was always roughly perpendicular to the confinement force direction, which reveals that the crack formation and propagation is mainly due to the equivalent tension stress at perpendicular direction under pressure. The result also shows that the mold confinement and contact state of crystals both could affect the crack distribution.

![Graph showing degree of crystal fracture versus pressure](image1.png)

**Fig. 7** Degree of crystal fracture versus pressure

![Images of slices under different pressures](image2.png)

**Fig. 8** Cracks under 5MPa pressure

### 3.2.4 Density

It is one of most important tasks of controlling and characterizing the density distribution during the explosive molding. In this paper, the gray value method is applied to study the pressing density distribution, assuming that the CT value (and thus the gray value) linearly depends on the sample densities. Our previous study also verified the good linearity of explosives and CT gray value (linearity $R > 0.99$) \cite{16,17}. Suppose the RDX crystals at initial state are homogeneous, with theoretical maximum density $\rho_{\text{crystals}}$. The initial packing volume is denoted by $V_0$, and packing density $\rho_{V_0}$, volume of each crystalline $V_{0j}$, average gray value of crystals $CT_0$, and average gray value of air $CT_{\text{air-0}}$. Cylinder density under certain pressure is denoted by $\rho_i$, volume $V_i$, average gray value of crystals $CT_i$, average gray value of air $CT_{\text{air-i}}$. Then we have:

$$\frac{\rho_i}{\rho_{V_0}} = \frac{CT_i - CT_{\text{air-i}}}{CT_0 - CT_{\text{air-0}}} \quad \text{(4)}$$

At initial state:

$$\rho_{V_0} V_0 = \rho_{\text{crystals}} \sum_{j=1}^n V_{0j} \quad \text{(5)}$$

Substitute formula (5) to formula (4):

$$\rho_i = \frac{CT_i - CT_{\text{air-i}}}{CT_0 - CT_{\text{air-0}}} \sum_{j=1}^n V_{0j} \rho_{\text{crystals}} \rho_{V_0} \quad \text{(6)}$$

Hence the quotient of density at different pressure can be written by:

$$\frac{\rho_i}{\rho_i} = \frac{CT_i - CT_{\text{air-1}}}{CT_2 - CT_{\text{air-2}}} \quad \text{(7)}$$

The relative density (with respected to mold density):

$$\frac{\rho_i}{\rho_{\text{model}}} = \frac{CT_i - CT_{\text{air-i}}}{CT_{\text{model-i}} - CT_{\text{air-i}}} \quad \text{(8)}$$
Finally, the bulk density ($\rho_v$) can be obtained by summing up slices:

$$\rho_v = \frac{1}{n} \sum_{j=1}^{n} \rho_j$$  \hspace{1cm} (9)

where the $j$ is the slice index, $n$ is the number of slices.

Density gradient between different parts is often concerned as its close relation with explosive performance. Densities of three parts (upper, middle and lower, as in Fig.3) are analyzed (top crack part of 150MPa case is abandoned). The analysis area is about 11.8mm$^2$. Fig.9a illustrates the $CT_{\text{CT air-i}}$ of three parts versus the pressure. From Fig.9, it can be seen that at initial state, CT gray value of bottom part was larger than other parts, and the middle part has the smallest gray value. When the pressure was increased, the gray value of three parts all grow nonlinearly and eventually become smooth. The gray value of upper part was still the largest, but the difference between lower part and middle part became negligible. According to the formula (7), the ratio of final density of three parts to initial density is illustrated in Fig.9b. The result shows that at low pressure, the increase of density was obvious at three parts, in which upper and middle part had roughly the same increase. When the pressure was increased, the density increase of middle part was larger than other parts evidently, which means at the initial loosely packing state, the density at lower part is a little higher. During the pressing process, the upper part has the largest density, but after molding, the middle part has the most density increase.

![Fig.9a](image1.png) ![Fig.9b](image2.png)

(a) CT gray value versus pressure  \hspace{0.5cm} (b) Ratio of final density to initial density

Fig.10 CT gray value and density ratio to initial state

Another set of octave points of sections are also analyzed, the area is about 2.75mm$^2$, the $CT_{\text{CT air-i}}$ of eight parts ($S_{ji}$) versus pressure is illustrated in Fig.10. As in Fig.10, at initial state, the parts had the smallest gray value and with the largest fluctuation. The gray value grew and the fluctuation decreased when the pressure was increased. According to formula (7), it means the density distribution of initial state had most fluctuation during the pressing process, while the density increased and the fluctuations decreased, but after molding under pressure of 150MPa, the radial density distribution tended to be uniform.

![Fig.10](image3.png) ![Fig.11](image4.png)

(a) CT gray value at octave points  \hspace{0.5cm} (b) Relative density distribution under different pressure
Fig. 11 illustrates the relative slice density (with respect to mold) under different pressure. Slice numbered 0 is the fixed end and maximum the pressing end. The bulk density and local density at different parts (50 slices at upper, middle and lower parts) are listed in Table 2. The result shows that slices of initial state had low density but roughly the same value at three parts, although the fluctuation of different slices was still obvious. When the pressure was increased, the density of all slices grew. The closer to the press end, the more the density increased. Fluctuation and gradient in density of different slices decreased. When the pressure was as high as to 150MPa, cracks emerged at the press end which led to a little density decrease. The final density at press end was higher than the fixed end, which means that higher pressure could be applied to obtain better density uniformity.

Table 2 Relative density distribution of three parts under different pressure

<table>
<thead>
<tr>
<th>Load Stress/MPa</th>
<th>0</th>
<th>5</th>
<th>20</th>
<th>40</th>
<th>50</th>
<th>150</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \rho_{v}/\rho_{model} )</td>
<td>0.687</td>
<td>0.844</td>
<td>0.895</td>
<td>0.940</td>
<td>0.987</td>
<td>1.034</td>
</tr>
<tr>
<td>( \rho_{1-50}/\rho_{model} )</td>
<td>0.683</td>
<td>0.738</td>
<td>0.841</td>
<td>0.905</td>
<td>0.960</td>
<td>1.006</td>
</tr>
<tr>
<td>( \rho_{middle+25}/\rho_{model} )</td>
<td>0.685</td>
<td>0.854</td>
<td>0.905</td>
<td>0.944</td>
<td>0.993</td>
<td>1.037</td>
</tr>
<tr>
<td>( \rho_{upper-50}/\rho_{model} )</td>
<td>0.709</td>
<td>0.912</td>
<td>0.923</td>
<td>0.956</td>
<td>0.989</td>
<td>1.051</td>
</tr>
</tbody>
</table>

4. Conclusions

It is proved to be high efficiency and strong capability to assess crystalline motion and fracture characterization in pressing by non-destructive technique of cone-beam µCT.

The results indicate the tendency of the displacement of RDX crystals is in nonlinearity, and the crystalline fracture is strongly influenced by the pressure and restricted by shape of the crystal mould. At initial loosely packing state, RDX crystals had higher density at bottom part. During the press process, the part closer to press end had higher density. The middle part had a larger density increase after molding.

It is also analyzed that the density distribution gradient is obvious along the pressing direction. Better density uniformity would be obtained under higher pressure. The movements of RDX crystals were mainly along the pressing direction. The crack direction was roughly perpendicular to confinement forces by neighbor crystals.

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References


