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Formulation of CL-20-Based Explosive Ink and Its Detonating Transfer Performance in Micro-Size Charge

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Abstract: Explosive ink in the micro-size charges of explosive circuits for energetic devices has attracted considerable interest. Herein, explosive ink with 90 wt.% of energetic solid was selected by testing the rheological properties of formulations with different CL-20 concentrations. Explosive ink suspensions with sub-micro CL-20 (2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane) as main explosive and fluorine-containing rubber/polyvinyl alcohol (PVA) emulsion as binder system were prepared. Direct ink writing (DIW) technology was used for the printing of the sample and for the loading of the explosive ink in a micro-size groove. The morphology and crystal type of the CL-20-based composite samples were characterized by SEM and XRD, and its detonating transfer performance in the micro-size groove were tested. Results show that the CL-20-based composite has a good molding effect, and the crystal type has not changed. The critical detonation thickness is around 11 μm at 1.0 mm fixed width in the wedge tests, and the critical detonation size (cross section) is about 0.3 mm × 0.3 mm. The detonation wave could continue to propagate around multiple 90° corners. The maximum corner turning ability of the detonation wave is 140° and the CL-20-based composite can detonate completely in a complex charge circuit.

Keywords: CL-20 · explosive ink · detonating transfer performance · corner turning ability · micro-size detonation

1 Introduction

Owing to the diversification and the complexity of modern war environment, the miniaturization of the weapon equipment, the complexity of the charge circuit, and the safety of the operation process have become of great concern [1]. The micro-fabrication method based on "layer-by-layer accumulation" of explosive inks by DIW technology has attracted the attention of some researchers in the field of energetic materials [2–11]. The technique has been used in printing pre-designed patterns quickly and effectively by means of computer-aided design and precision machinery. Two methods are usually used in the fabrication of energetic micro-devices by using DIW technology. In the first method, a piezoelectric nozzle with minimal caliber is used to spray all-liquid explosive liquid ink one by one at a predetermined position. Then, the energetic composites are prepared by solvent volatilization of printed materials [7–10]. In the second method, an explosive ink suspension is extruded out of the needle with the help of an external force to form a predetermined pattern. An energetic composite is formed by layer-by-layer accumulation. Most researchers are interested in studying the crystal type, thermal decomposition performance, rheological properties, and critical detonation size of a formulation. Their studies undoubtedly provide a good reference for the application of explosive ink in the micro-size charges of explosive circuits but remain inadequate. As is well known, explosive circuits have been widely used in aimed warheads and shaped charge warheads, due to their small size and reliable function. Owing to the miniaturization of weapon systems, the small size of explosive circuits poses challenges to the traditional charge methods and processes, such as the pressing and casting methods. DIW technology of explosive ink can solve these problems easily. Nevertheless, in complex charge circuits, the corner turning ability of the detonation wave is a problem that should to be studied thoroughly. It should be regarded as an index for evaluating the detonating transfer performance of explosives and is as important as the critical detonation size.

In 1969, Silvia and Ramay [12] proposed the corner turning effect, which can contribute to the realization of an explosive logic circuit. This design principle provides a new way of thinking for the application of explosives in logic and intelligence. Thus, designing a suitable test method for the corner turning ability of the detonation wave in a micro-size groove charge is very important. Moreover, increasing the content of the main explosive in the formulation is a suitable

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method for ensuring that the explosive ink charged in a micro-size groove has enough explosion power. However, this method changes the properties of an explosive ink formula, such as rheology and formability. Especially, the viscosity of explosive ink suspensions increases, and extruding ink from a nozzle becomes difficult. In our previous work [6], CL-20-based explosive ink (CL-20 88% concentration) was prepared, which was composed of a Viton A (vinylidene hexafluoropropene copolymer)/PVA emulsion as the binder system and the service life of the emulsion was only 60 h. The critical detonation thickness of this composite was approximately 0.17 mm, and the fixed groove width was 1.0 mm in the wedge test. Given that the service life of a binder emulsion system is short, the contents of each component and the conditions of the preparation process of the binder emulsion were systematically optimized, and the static state stability of the emulsion was greatly improved from 60 h to 360 h in some of our works [13].

In this study, the optimized emulsion was used as binder system for the preparation of explosive inks with different CL-20 contents. Then, the best explosive ink formulation was selected by testing the suspension rheological properties, and the inner structures and crystal types of the printed samples were characterized. Lastly, the detonating transfer performance of CL-20-based composite in a micro-size groove was measurement and analyzed.

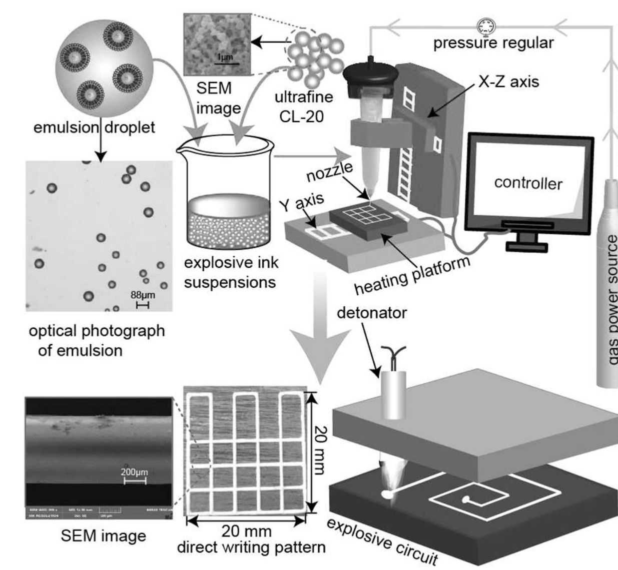
2 Experimental Section

2.1 Suspension Preparation and Rheological Measurements

An emulsion binder system of fluorine-containing rubber/PVA was prepared according to a previously described method [13]. Then, an appropriate amount of sub-micro CL-20 powder was added to deionized water. The mixture was subjected to ultrasonic stirring at 20 kHz for about 10 min, for the complete dispersion of the sub-micro CL-20 particles. A small amount of emulsion was added to CL-20, and the mixture was stirred for about 3 h at 60r·min^{-1}. Explosive ink suspensions with a weight percent of 84 wt.%, 86 wt.%, 88 wt.%, 90 wt.%, 92 wt.% and 94 wt.% (the weight of the solids) were prepared. The rheological behavior of the six kinds of explosive inks with different CL-20 concentrations was tested with an R/S rheometer (Brookfield Engineering Laboratories, Inc., Middleboro, MA, USA). The test conditions were as follows: shear rate: 1–100 s^{-1}, room temperature: 25 °C, measuring points: 60 and the measuring time: 60s.

2.2 Performance Characterization and Testing

The best formulation was selected based on the rheological parameters of the explosive ink suspensions. The surface and inner structure (the cross section of sample) of the printed samples was observed by a TESCAN Mira 3 Field Emission Scanning Electron Microscope (FE-SEM), and the crystal types were distinguished by a DX-2700 X-ray diffraction instrument (XRD) (Dandong HaoYuan Instrument Co., Ltd).The density of the composite was characterized five times with a MZ-220SD electronic densimeter (Shenzhen force Tatsu letter instrument Co., Ltd.), and the mean value was obtained. The platform of the DIW was consistent with that in our previous work [6]. That is, the deposition velocity varied according to the nozzle diameter and the gas pressure of the explosive ink suspensions. In the present work, syringes filled with suspensions were loaded into a computer-controlled robocasting machine, and the suspension was dispensed through cylindrical nozzles of 0.21–0.50 mm in diameter at a rate of 1–10 mm/s onto a moving plate at a gas pressure of 0.05–0.35 MPa. Nozzle height, nozzle angle, and temperature console were 0.3 mm, 90°, and 50 °C, respectively. DIW technology was used in the layer-by-layer loading of the explosive ink in a micro-size groove, and the printed sample was dried in a constant temperature water bath oven (55 °C) for 12 h. The resulting CL-20-based composite was characterized by XRD, SEM, and detonating transfer performance test. The process flow diagram of direct ink writing is shown in Scheme 1.



Scheme 1. Process flow diagram of direct ink writing.

3 Results and Discussion

3.1 Rheological Behavior of the Explosive Ink

Figure 1 shows the viscosity curves of the explosive ink suspensions with various CL-20 concentrations as a function of the shear rate. The viscosity obviously decreased at increased shear rate. The suspensions showed a shear-thin-

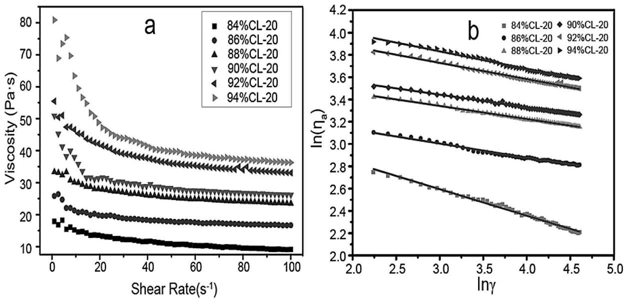


Figure 1. Rheological behavior of explosive ink suspensions with different CL-20 content.

ning behavior in the shear-rate range of 1–100 s^{-1}, and the relationship between apparent viscosity and shear rate was nonlinear. Hence, the explosive ink suspensions belonged to the pseudoplastic fluids, which can be described by the Ostwald-de Waele energy power law equation [14–17]:

$$\eta_a = K \times \gamma^{n-1} \tag{1}$$

Here η_a is the apparent viscosity, Pa·s; K is consistency coefficient, Pa·s^n; γ is shear rate, s^{-1}; n is fluidity index. The rheological data were also fitted to this model for the calculation of fluidity index (n) and consistency coefficient (K) by Equation 2.

$$\ln\eta_a = \ln K + (n\text{-}1)\ln\gamma \tag{2}$$

The fitting data are the apparent viscosity corresponding to the shear rate of 10–100 s^{-1} (because suspensions in the shear rate of 1–10 s^{-1} are not steady state flow).The results are shown in Figure 1 b. The consistency coefficient *K* and fluidity index n can be calculated, according to the slope and intercept of the fitting line. The parameters of the fitting lines are shown in Table 1.

In Table 1, the *K* values increased with suspension concentration. The n value of the suspension with 90 wt.% CL-20 reached a maximum value is 0.887, which is close to 1, showing a typical Newtonian fluid behavior. Plastic flow behavior, Newtonian flow behavior, and dilatant flow behavior corresponded to three different ranges: $n > 1$, $n = 1$, and $n < 1$, respectively [18]. The closer the n value is to 1, the smaller the degree of fluid deviation from Newtonian fluid and the stronger the ability of the fluid to resist external disturbances. Meanwhile, *K* rapidly increases when the CL-20 content is greater than 90 wt.%. Increasing the *K* value increases the viscosity and decreases the fluidity of the explosive ink suspension, thus increasing the difficulty of extruding explosive ink out of the nozzle. Thus, explosive ink with 90 wt.% energetic solids is often selected for micro-size charge.

3.2 SEM and XRD Analysis of the Composite Sample

The surface and internal structure of the printed sample was observed by SEM. In Figures 2 a1, a2, and a3, some holes were observed on the surface of the sample at high magnification. One possible reason for this phenomenon is the evaporation of solvents during curing. The cross-section (prepared by free break) of the sample was used in the observation of the inner structure of the composite. Figure 2 b1 shows the cross-section of the sample, which was formed by the layer-by-layer accumulation of explosive ink.

As shown in Figures 2 b2 and b3, the printed sample has a satisfactory molding effect, and no obvious pores were observed in the inner part of the sample. Additionally, the consistency between the accumulated layers of the explosive ink was good. The density of the CL-20-based composite was 1.65 $g\cdot cm^{-3}$, and the theoretical maximum density (TMD) was 1.947 $g\cdot cm^{-3}$.

CL-20 is a polymorphic substance with four common crystal forms, of which ε-CL-20 has the most optimal comprehensive performance [19]. Sub-micro CL-20 was obtained by the mechanical milling method, which is a green refining method that does not change the crystal type of the original material [20]. The crystal type of CL-20 in the composite samples was determined by XRD at angles from 5° to 50°, a step angle of 0.03, and a test temperature of 20 °C to distinguish the crystal type of CL-20 in composite samples. Figure 3 shows that the positions of the three strong characteristic peaks of the raw CL-20 (produced by Liaoning Qing yang Special Chemical Co., Ltd), sub-micro CL-20 and CL-20-based composite are basically the same, and their diffraction angles at 12.6°, 13.8°, and 30.3° agree

Table 1. Parameters of fitting lines and calculated values of K and n.

CL-20 (wt.%)	Intercept (ink)	Error	K	Slope (n-1)	Error	n	R2
84	3.307	0.010	27.30	-0.238	0.003	0.762	0.994
86	3.383	0.008	29.46	-0.127	0.002	0.873	0.996
88	3.696	0.004	40.29	-0.118	0.001	0.882	0.996
90	3.783	0.005	43.95	-0.113	0.001	0.887	0.994
92	4.167	0.007	64.52	-0.146	0.002	0.854	0.991
94	4.305	0.009	74.07	-0.157	0.002	0.843	0.990

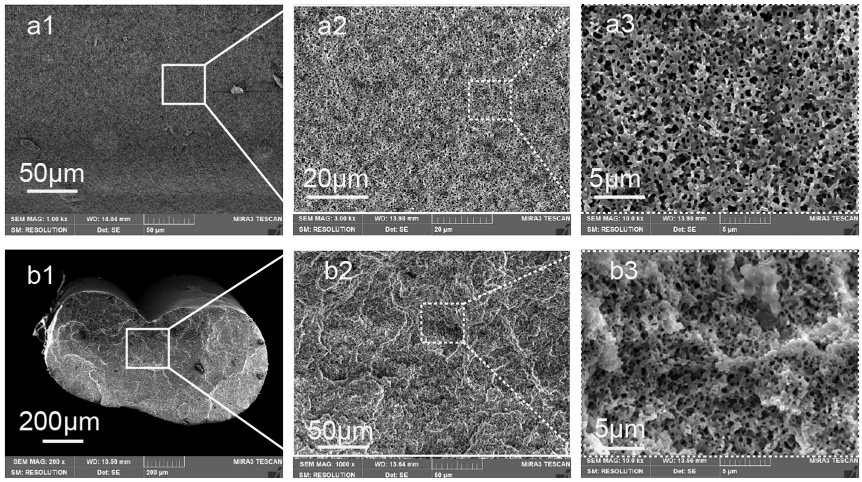


Figure 2. SEM photos of surface (a1, a2, a3) and cross-section (b1, b2, b3) of CL-20 based composite after curing.

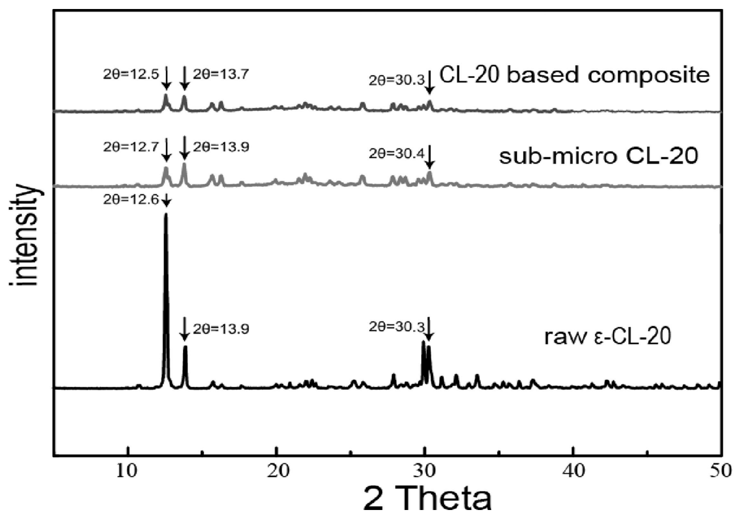


Figure 3. X-ray diffraction spectra of raw CL-20, sub-micro CL-20 and CL-20-based composite.

with the standard PDF card (00-050-2045) of ε-CL-20. This result also agrees with our previous work [6,13].

3.3 Critical Detonation Thickness and Cross-Section Size

The critical detonation thickness of the CL-20-base composite in the wedge groove was tested with a wedge-shaped charge method. Substrates with micro-size grooves were designed and configured according to the machining standards of China The test method refers to the wedge test standard in MIL-STP-1751 A. Explosive ink was loaded in a wedge shape groove (with fixed width, maximum depth, and length of 1.0, 3.0 and 100 mm, respectively) by DIW technology. Then, the printed sample was dried in a constant temperature water bath oven (55 °C) for 12 h. The obtained CL-20-based composite was subjected to wedge test. As shown in Figure 4 c, the length of the explosive trace on the aluminum plate was 99.64 mm. The critical thickness of detonation can be calculated by equation $d_c = C \times (A-B)/A$, where A is the length of the wedge groove (100 mm), B is the length of the explosive trace on aluminum plate, and C is the depth of the bottom (3.0 mm), as shown in Figure 4 a. The critical detonation thickness of the composite can be calculated, and the value is around 0.011 mm, which means that the critical detonation thickness of the CL-20-based composite is 0.011 mm, and the groove width was 1.0 mm.

In the critical thickness size experiment, a propagating detonation typically does not fail immediately when the charge dimensions drop below the "critical thickness," and instead "tunnels" for some distance in the wedge tests. Therefore, the value of the explosive traces was larger than the actual one, and 11 microns was smaller than the actual

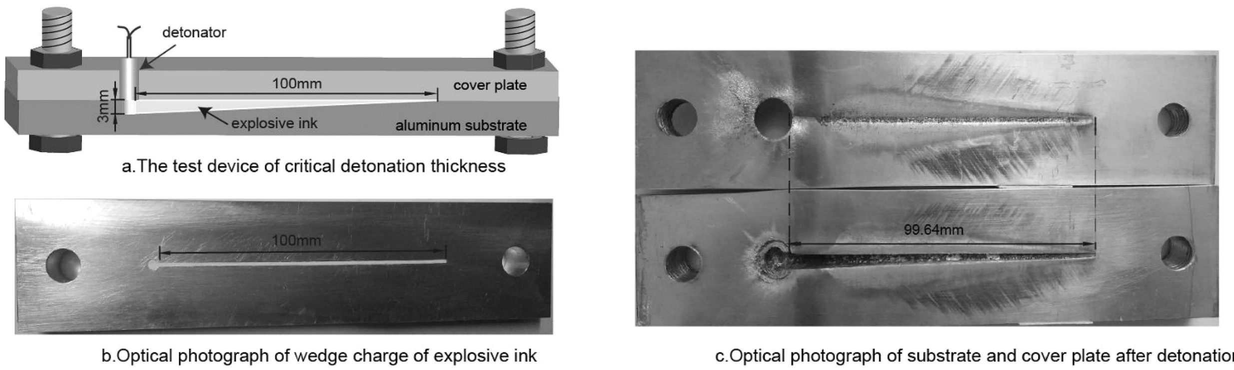


Figure 4. The measurement of critical detonation thickness of explosive Ink.

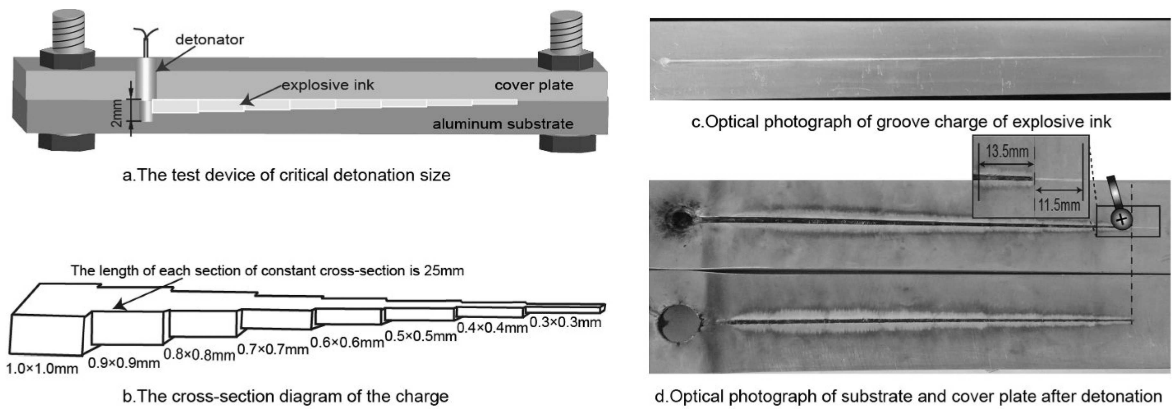


Figure 5. The measurement of critical detonation size of explosive Ink.

critical thickness in such conditions. Although the wedge test has some limitations regarding to the quantification of real critical detonation thickness, it is still the most commonly method for measuring critical detonation thickness. Furthermore, the result of the experiment qualitatively reflects the detonating transfer capacity of an explosive and can be used in researching the detonation performance of an explosive ink composite. Thus, these results have an important reference value.

As the uncertainty of the character and the purpose of the explosive circuit, the structure of an explosive element is complex and diverse. Thus, the critical detonation thickness of an explosive ink in small scale hardly satisfies the requirements for a practical application. For this reason, the critical size (cross section) of the detonation was tested with a method using the critical explosion size. The test device is shown in Figure 5 a. The square cross-section of the charge declined stepwise. The sizes of the cross-section were 1.0 mm×1.0 mm, 0.9 mm×0.9 mm, 0.8 mm×0.8 mm, 0.7 mm×0.7 mm, 0.6 mm×0.6 mm, 0.5 mm×0.5 mm, 0.4 mm×0.4 mm, and 0.3 mm×0.3 mm, respectively. The length of each section of the constant cross-section was 25 mm, as shown in Figure 5 b. The explosive ink was loaded in the square groove of this size by DIW technology. As shown in Figure 5 d, the charge cross-section size of explosive ink in the groove was between 1.0 mm×1.0 mm and 0.4 mm×0.4 mm, and detonation propagation was stable. When the detonation propagated to the charge cross-section of 0.3 mm×0.3 mm, it continued to propagate at approximately 13.5 mm, before extinguishing. Hence, we arrived at the following conclusion: the critical detonation size (cross-section) of the CL-20-based composite was about 0.3 mm×0.3 mm.

3.4 Corner Turning Ability of Detonation Wave

The corner turning ability of the detonation wave is an important parameter for measuring the detonating transfer performance of an explosive. In a classic example of a corner turning test, a small diameter explosive is detonated, and the detonation wave propagates into a larger diameter explosive as described by Los Alamos' Mushroom test [21]. Many researchers are interested in it and have done a lot of work [22–26]. The corner turn ability of the detonation wave is a key factor for determining whether an explosive can be applied in an explosive circuit. According to the detonation theory [27], when the detonation wave reaches the

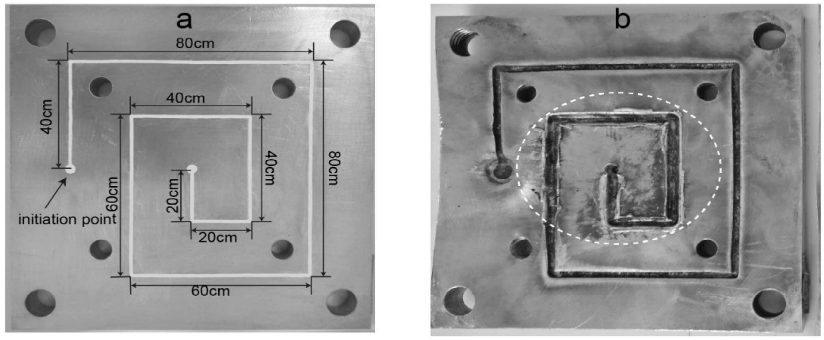


Figure 6. (a) Optical image of explosive circuit charges with multiple 90° corner before detonation; (b) Optical image of charge substrate after detonation.

corner, a cluster of central expansion waves propagates from the corner, weakens the intensity of the detonation wave and reduces the velocity. In the process of a diffraction detonation wave, the area of the front of the shock wave increases suddenly, and the intensity decreases, thus the chemical reaction of the lower explosive is retarded, and the reaction zone lengthens. Under specific conditions, detonation can be reformed again. However, when the energy of the diffraction detonation wave is inadequate to initiate detonation, it acts as a preloading effect on the explosive, and reduces the sensitivity of the explosive due to the density increase in the unexploded area, the so-called "dead zone" [21,27,28]. Based on this idea, we designed an explosive circuit with multiple 90° angles. The charge circuit formed a "spiral shape" as shown in Figure 6. In this experiment, the DIW technology was utilized in the loading of the explosive ink in the groove circuit; the size of the groove was 1.0 mm × 1.0 mm. After curing, detonation was started with number 8 detonators. A strong constraint between the substrate and the cover plate was found.

As shown in Figure 6, the CL-20-based composite can propagate the detonation reliably in such an explosive circuit. Thus, the detonation wave continuously propagated after turning around the 90° corner with partial energy losses. Notably, the explosive trace in the center of the "spiral explosive circuit" was much more obvious than the explosive trace around it, as can be clearly observed in the circle in Figure 6 b. One possible reason is that the restraint intensity in the center of the explosive circuit was greater than that in the surrounding area, which causes the detonation wave to lose less energy during propagation, the explosion power is high and making the width of the groove expands more obvious.

The spiral explosive circuit experiments showed that the detonation wave formed by the explosive ink continuously propagated after corner turning at 90°. However, this result is insufficient for the explosive ink applied to the micro-size charge of the explosive circuit, because most explosive elements have complex charge structures. On this basis, an explosive circuit with different angles and a 1.0 mm × 1.0 mm charge groove (shown in Figure 7 a) was designed and used for testing the maximum corner turning ability of the detonation wave. The results are shown in Figure 7 b.

When the corner turning angle θ is 0, the detonation wave propagates along a straight line, and the energy losses of the detonation wave increase with θ. The initiation ability of a diffraction detonation wave weakens gradually, at increasing angle θ and is extinguished at a certain value. As shown in Figure 7 b, the CL-20-based composite detonated successfully when the corner turning angle was less than 140° and left an obvious explosive trace on the cover plate. When the corner turning angle was more than 140° the CL-20-based composite failed to detonate and no explosive trace was observed on the cover plate, as observed in the circles in Figure 7 b. Thus, we can conclude that the maximum corner turning ability of the detonation wave is 140° for the CL-20-based composite. This result is based on the size of charge cross-section of 1.0 mm × 1.0 mm. When the size of charge cross-section tended to infinity, the detonation appeared as a one-dimensional plane detonation, and corner turning problem of detonation was non-existent.

3.5 Application of Explosive Ink in Micro-Size Charge Circuit

According to the result of the corner turning ability of the detonation wave, the detonation wave produced by the CL-20-based composite continuously propagated turning around a large angle. This result means the explosive ink can be applied to a complex explosive circuit. In Figure 8, the explosive ink was loaded in an aluminium plate with a complex groove circuit. The cross-section size of the groove was 0.9 mm × 0.9 mm. As shown in Figure 8 b, the CL-20-based composite can completely detonate through a groove circuit with different corners between 0° and 140°. The result demonstrates that the explosive ink can fully detonate in a complex, multi-angle groove circuit. This result

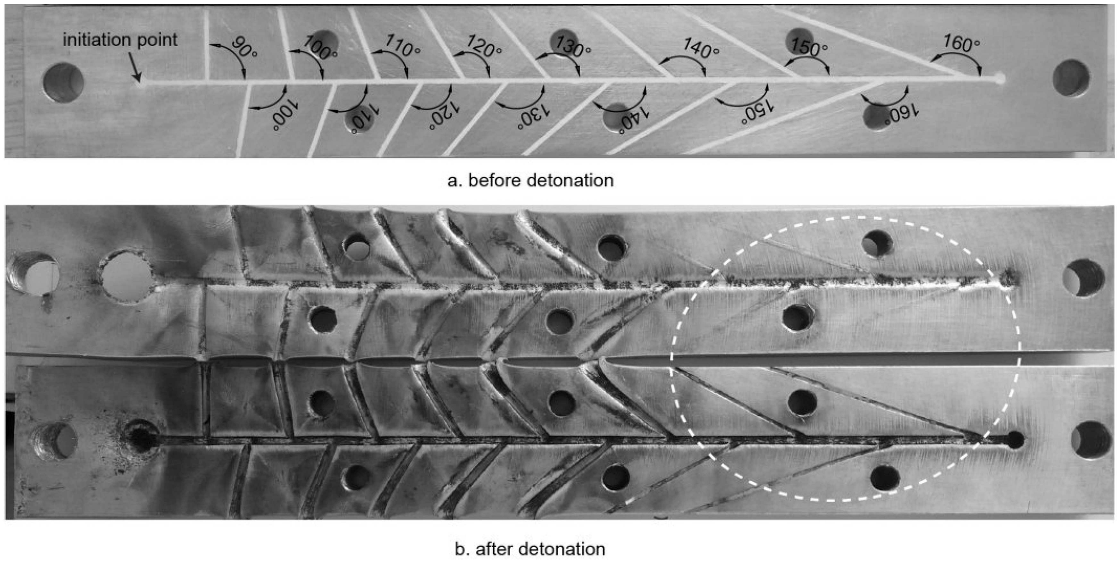


Figure 7. The measurement of corner turning ability of detonation wave.

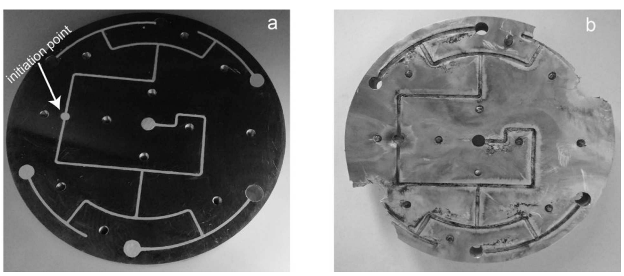


Figure 8. The measurement of detonation performance of explosive ink in small-size explosive circuit.

can serve as a reference for the detonating transfer performance of explosives in micro-size charges.

4 Conclusions

We selected a formulation of CL-20-based explosive ink with 90% energetic solid by testing the rheological properties of explosive ink with different CL-20 concentrations. A 90 wt.% CL-20-based explosive ink was loaded in the micro-size groove with a DIW technology, and the performance of the printed samples was characterized. The SEM images illustrated that a satisfactory molding effect of the composite was achieved. The XRD patterns showed that the type of CL-20 does not change. In the study of the micro-size charge of the CL-20 composite, it shows a good detonating transfer performance of the CL-20-based composite in a micro-size charge. The critical detonation thickness was approximately 1.0 mm×0.011 mm in the wedge tests, and the critical detonation size was 0.3 mm×0.3 mm in the cross-sectional gradient decline tests. The maximum corner turning ability of the detonation wave was 140°. The wave can successfully propagate in a micro-size charge circuit with multiple 90° corners, and even in more complex explosive circuits. This study may serve as a reference for further research on suspended explosive inks and provide a good technical support for the application of explosive inks in micro-size explosive circuits.

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