

Electrostatic discharge sensitivity of titanium potassium perchlorate at varying densities

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Abstract

Titanium potassium perchlorate (TKP) powders subjected to an electrostatic discharge (ESD) have been shown to be sensitive to ignition from a standard human body model waveform. Two particle sizes with differing morphologies of titanium at varying loading densities were ESD insulted to determine their ignition sensitivity. By using a partially confined geometry for the TKP powders the configuration more closely relates to that of an ignitor during production. This configuration shows a trend in ignition related to the density variation, from loose powder to ~ 2.5 g/cc (densities commonly found in ignitors) and the discharge voltage from the standard human body model waveform MIL-STD-331 (1), up to 25 kV. The results show a difference in sensitivities between the two particle sizes of TKP powders tested. This leads to an understanding of how pressed powder density, or the encompassed void size based on the particle morphology, affects the spark sensitivity. Pressed powders were cross-sectioned and imaged to obtain quantitative microstructural data. A correlation can be drawn from the reaction of the powder to the available oxygen in the void space, which agrees with Collins (2) findings.

1 Introduction

The production of pyrotechnic devices have consistently been plagued with unintentional initiation. A main source of unintentional initiation corresponding to pyrotechnic powder is ESD. This has led many manufacturers to adopt, modify, or create standards in order to assess the level of sensitivity of a powder to an ESD, with some of them here as reference: MIL-STD-1751 (3), Carlson and Wood (4), STANAG 4490 (5), Westgate et al. (6). Standards typically define the sensitivity level by comparing sensitivity of a loose powder to a reference energetic. The test methods used in the standards also deviate from one another, but most stem from a version of the ABL Model 150 ESD Sensitivity Tester (Hercules Aerospace Company, Allegany Ballistics Laboratory, Rocket Center, West Virginia).

The sensitivity of pyrotechnics has led to large amounts of research and development on variations of pyrotechnics that have characteristics that are considered insensitive. A pyrotechnic of interest here is TKP, an energetic that has been used for over 80 years (7). A variation that was formulated to control the initiation characteristics through variations in the hydride stoichiometry was titanium subhydride potassium perchlorate (THKP) (8). While this formulation does desensitize the pyrotechnic to ESD initiation, it also makes intentional initiation more difficult. ESD sensitivity not only is influenced by the formulation, but may be affected by particle surface area (9), inferred from Collins et al. (10) work with available oxygen.

An aspect of ESD sensitivity that has not received a lot of attention is the effect of density on spark ignition. In order to understand this relationship, a charge cavity of powder is pressed to a dead stop (for column height consistency) and ESD insulted using a tester similar to the ABL

Model 150 ESD Sensitivity Tester. The test follows MIL-STD-331 (1) for the standard human body waveform, with the parameters of the base circuit listed in Table 1. Herein two variations of TKP are studied. One TKP powder (TKP-I), is composed of 33% Ti and 67% KClO_4 by weight. TKP-I has a Ti particle size of $13\pm 3\mu\text{m}$ at the 50th percentile and a minimum surface area of $3.5\text{ m}^2/\text{g}$. The other powder (TKP-II), has 41% Ti and 59% KClO_4 by weight. TKP-II has a Ti particle size of $20\pm 4\mu\text{m}$ at the 50th percentile and a surface area range of 0.20-0.50 m^2/g . The KClO_4 in both powders are specified to have a maximum particle size of 14 μm at the 50th percentile.

Table 1: Specification for experimental setup using MIL-STD-331 (1).

	MIL-STD-331
Maximum Voltage (kV)	25
Resistance (Ω)	500
Capacitance (pF)	500
Inductance (μH)	5

2 Experimental Setup

The two TKP powders were tested in a commercial boom box/firing system, Model 931 Firing Test System, with the optional powder electrode assembly from electro-tech systems, inc. (a stationary version of ABL Model 150 ESD Sensitivity Tester). The powder test electrode (PTE) assembly consists of a needle electrode, an adjustable height grounded base with locking ring, plus stainless steel plug counter-bored to hold the powder cup, shown in Figure 1(a). The powder cup is made up of two pieces, a dielectric plastic (high density polyethylene) washer to confine the sides, and an aluminum base with a small nipple in the center to serve as a conductive path. TKP powders were pressed and tested within the charge cavity ($\varnothing 0.150\times 0.066\text{ in.}$), shown in a cutaway view in Figure 1(b).

Prior to testing of any powder a ringdown is performed to verify the waveform characteristics meet expectations. To perform a ringdown an empty charge cavity is adjusted on the base to be positioned slightly below the electrode tip (not in contact), and then the capacitor is discharged. Figure 1(c) shows a typical ringdown of the entire assembly when the capacitor is charged to its maximum 25 kV and recorded at 200 MHz. This measurement was taken via an in-line $2\ \Omega$ current viewing resistor (CVR).

Testing on TKP powders is performed similarly to the ringdowns: the powder is positioned slightly below the electrode tip, the capacitor is charged to a specified voltage, then the powder is insulated with a single ESD. A current viewing transformer (CVT) is used to monitor the ESD pulse since it is less intrusive to the system than a CVR.

3 Results and Discussion

The ESD tests were performed on the two variations of TKP powders, pressed at densities ranging from $\sim 0.68\text{ g/cc}$ (loose) to $\sim 2.52\text{ g/cc}$. This density range for TKP-I and -II corresponds to 23%–85% theoretical maximum density (TMD) and 22%–82% TMD, respectively. The results of the tests are considered either a “Go” when all of the powder in the charge cavity ignites and is consumed or a “No-Go” otherwise. Figure 2 shows the outcome of comparing ESD voltage versus pressed powder density.

There are a few interesting observations from the ESD testing shown in Figure 2. The TKP-II while at a loose density ignites at 20 kV, it progressively becomes more sensitive up to $\sim 1.50\text{ g/cc}$

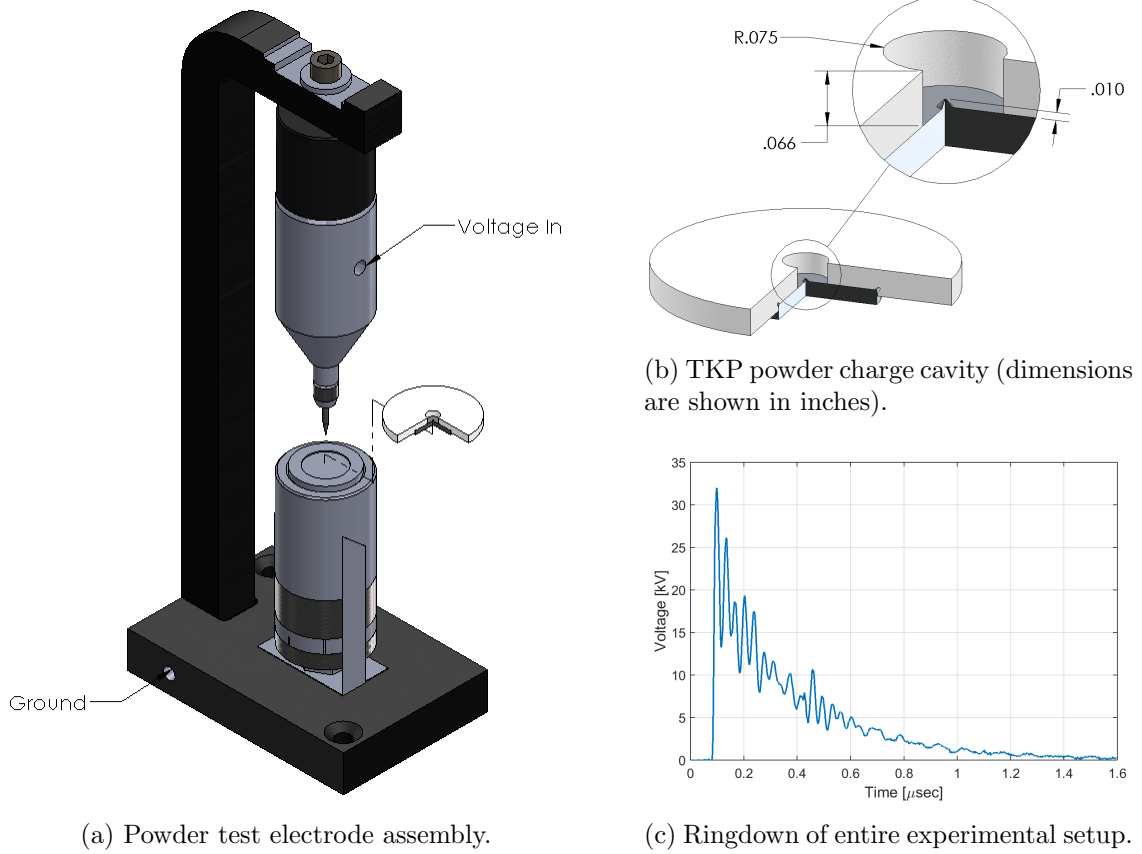


Figure 1: Experimental setup of powder test electrode (PTE) assembly (a), the needle probe tip is stationary while the base is adjustable for height accommodations. PTE base is modified to hold the powder charge cavity. The open to atmosphere, powder charge cavity (b) (shown in a cutaway view), is confined on the sides with a dielectric and on the bottom with a conductive aluminum plate (dimensions are shown in inches). (c) shows a voltage ringdown of the MIL-STD-331 (1) electrical circuit across the resistor in a current viewing resistor (multiplied by the resistance) and PTE assembly in series.

igniting at 7 kV. At higher densities, TKP-II decreases sensitivity and does not ignite with a maximum discharge voltage 25 kV beyond ~ 2.04 g/cc. TKP-I on the other hand does not show a broad span of sensitivity variation with density. TKP-I at loose density is more sensitive than TKP-II, igniting at voltages as low as 4 kV. There is a slight sensitivity increase at ~ 1.00 g/cc, ignition occurred as low as 3 kV. There is a steep drop in sensitivity of TKP-I and it is unable to ignite with the maximum discharge voltage of 25 kV beyond ~ 1.53 g/cc. There seems to be regions of high sensitivity at moderate densities for both powders, and then at higher densities they are unable to initiate.

To investigate reasons for the trends seen in the ESD sensitivity tests, the powder morphologies were imaged and analyzed. In order to image the pressed powder beds, cross-sections were created using an ion beam milling technique. The cross-sectioned powders were then imaged using scanning electron microscopy (SEM). TKP-I and -II samples were cross-sectioned and imaged at a high density ~ 2 g/cc. A euclidean distance of the void to a particle, a transform of distance of each pixel in the void to the nearest interface (Ti or KClO_4), is then extracted from the images in order to see if there is a correlation in void size and the sensitivity of the powder. Figure 3 shows TKP-I and -II cross-sections and euclidean distance counts of the images.

The two powders ESD tested, TKP-I and -II, have the same KClO_4 particles, yet the Ti particles have a surface area 10 times larger in the TKP-I. In the SEM images (Figure 3) the Ti is slightly smaller than the KClO_4 in -II, and in the -I powder is quite a bit smaller and easier to

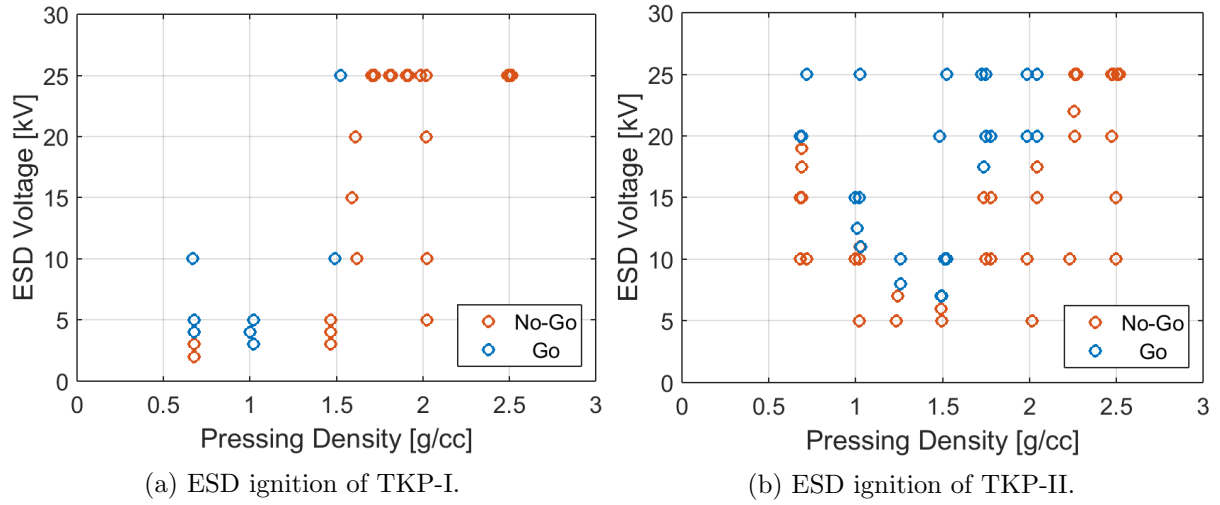


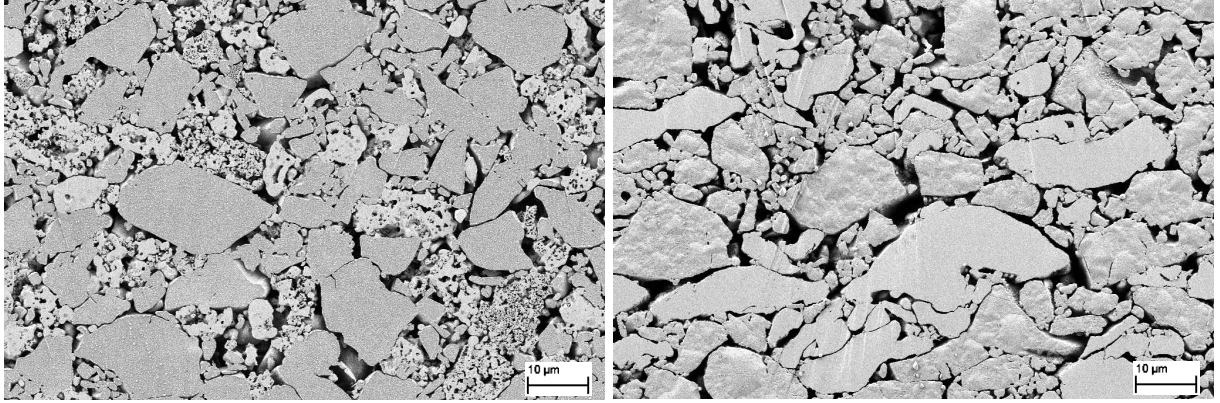
Figure 2: Single pulse ESD on -I (a) and -II (b), "Go" and "No-Go" data are recorded at various densities and voltages.

recognize due to the porous morphology. The semi-quantitative void sizes of the TKP-I and TKP-II powder are shown through the euclidean distance maps in Figure 3(c). The euclidean distances for -I and -II show a similar trend, which corresponds to their comparable ESD sensitivities at this pressing density (refer to Figure 2). As the pressing density is lowered there is expected to be a variation in the euclidean distance map of the voids from the high density case shown in Figure 3.

Low density (~ 1 g/cc) TKP-I and -II produced challenges while attempting to ion beam mill for SEM images. Due to the low density, particles tended to shift and not cross-section cleanly. Rather than ion beam milling, micro-computed tomography (micro-CT) scans were created of low density TKP-I and -II, shown in Figure 4. The current scans do not have the resolution to make quantitative results of euclidean distances of the voids, however, this technique shows promise in future runs of being able to extract euclidean distance of void/particle interface. With longer exposure times and higher magnification, the micro-CT data will have resolution similar to that of the SEM images shown in Figure 3, making quantitative 3D analysis possible.

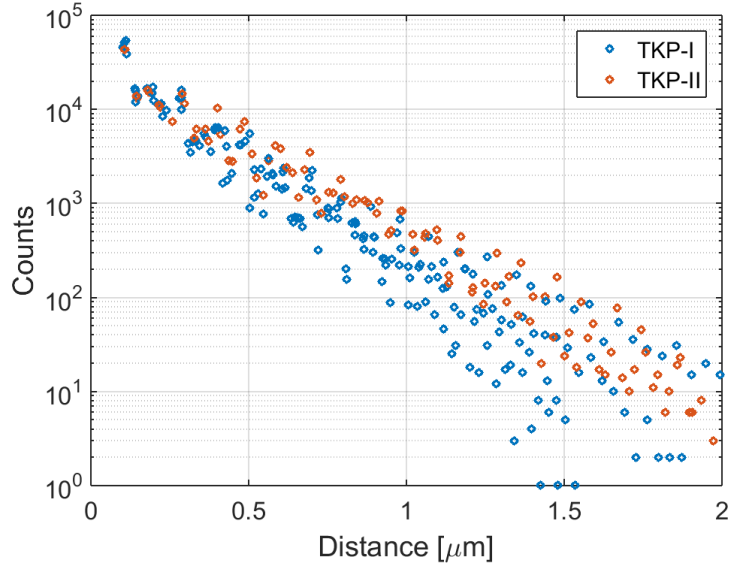
4 Conclusions

It is shown from the ESD tests conducted that there is a change in sensitivity based on the pressed powder density. There are regions of high sensitivity at moderate densities for both powders, and then at higher densities they are unable to initiate with the parameters of MIL-STD-331 (1). ESD penetration has been theorized to follow a percolation effect into the powder bed (11). This, along with available oxygen content examined by Collins (2), may result in the sensitivity variations shown. By using cross-sectioning techniques, ion beam milling with SEM and micro-CT, we were able to image particles and voids within a pressed powder cavity. Using image analysis, void size was extracted from the cross-sectioned images, based on the euclidean distance to a particle interface. There were no significant differences in the euclidean distance maps for TKP-I and -II, which corresponded to their similar ESD sensitivities at that density. Low density pressed powders were imaged via micro-CT, with promising results for future analysis of euclidean distance mapping.



(a) TKP-I cross-section of pressed powder.

(b) TKP-II cross section of pressed powder.



(c) Counts of euclidean void distance to nearest interface.

Figure 3: Pressed powder bed cross-sections created using ion beam milling. The sections of TKP-I (a) and -II (b) are imaged with SEM. (a) and (b) are high density pressings (~ 2 g/cc). In the images, KClO_4 are the larger particles and Ti is smaller. The Ti in the -I is easier to pick out due to the small size and porous morphology. (c) shows euclidean distances of TKP-I and -II on a semi-log plot. TKP-I were analyzed from four images and -II were analyzed from two images, each having $100\text{ }\mu\text{m}$ wide field of view.

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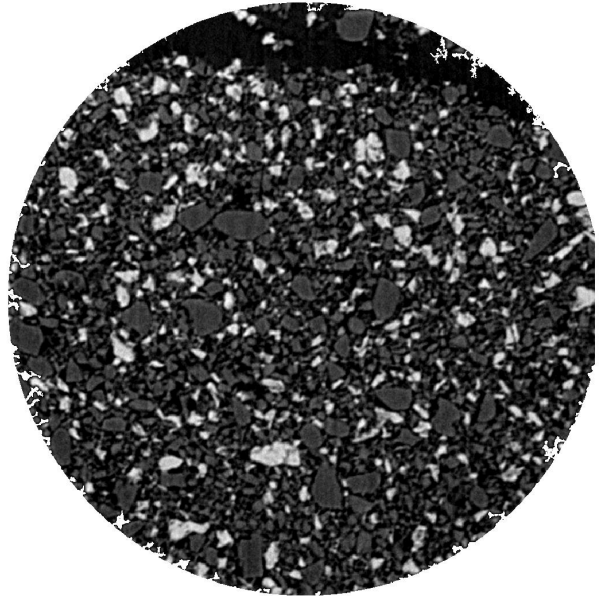
(a) Micro-CT scan of TKP-I.



(b) Micro-CT scan of TKP-II.



(c) Micro-CT slice of TKP-I.



(d) Micro-CT slice of TKP-II.

Figure 4: Micro-CT scans of TKP-I, (a), and TKP-II, (b), are low density pressings (~ 1 g/cc). Cylindrical volumes shown are $\sim \varnothing 0.75 \times 0.75 \mu\text{m}$ high. Slice of both TKP-I and TKP-II are shown in (c) and (d), respectively.

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