ELECTROSTATIC DISCHARGE EFFECTS ON RESISTANCE OF TITANIUM POTASSIUM PERCHLORATE IGNITORS

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ABSTRACT

Electrostatic discharge (ESD) applied to titanium potassium perchlorate (TKP) loaded ignitors resulted in decreasing electrical resistance measured across the bridgewire, degrading the nominally 1-Ω hot-wire ignitor by as much as 33%. Such changes in resistance effect functional performance and potentially 1A/1W no fire safety. Previous work [1] identified two mechanisms responsibly for the ESD induced resistance changes in the TKP ignitors. This work investigates ESD induced resistance change by examining differing TKP mix ratios, densities, powder geometries, titanium particle size distributions and morphologies. Results were compared against hot-wire ignitors with different TKP volumes and pin spacing with each having a spark gap designed to prevent ESD from flowing through the bridgewire and affecting the pyrotechnic. MIL-STD-331C ESD pulse waveforms were applied varying the amplitude as well as the number of insults applied to the test ignitor.

1 Introduction

As part of the safety theme, pyrotechnic hot-wire devices are designed and tested to withstand electrostatic discharge (ESD), particularly as applied by the human body. These devices (ignitors, initiators, and actuators) include design features such as sparks gaps and electrical isolation to help prevent inadvertent ignition by limiting unintended electrical stimulus to the pressed pyrotechnic powder. Emphasis is placed on the device's ability to reliably operate throughout its design life. Devices are submitted to environmental conditions including ESD insults from simulated repeated (lifetime) handling that may occur during assembly and disassembly. As part of production testing, it is not uncommon to apply ESD pulses between the device's pins (shorted together) and the metal case or pin-to-pin. MIL-STD-331C [2] is a standard test method for conducting environmental tests on fuzes (e.g. ignitors), and can include multiple ESD hits to the test article as part of the environmental test sequence. These tests should be non-degrading to deliver a viable ignitor for its intended use.

Hot-wire devices are commonly built such that the resistance measured between the pins across the bridgewire (BW) is 1 Ω. Resistance measurements during product testing and acceptance provide another means to identify faulty or damaged product by confirming resistance remains within tolerance.

Ignitor degradation from ESD induced damage, documented by Hingorani [3] for titanium subhydride potassium perchlorate (THKP) and titanium potassium perchlorate (TKP) hot-wire devices following multiple ESD pulses, resulted in failure to meet all-fire (functional) and no-fire (safety) requirements. Post-ESD examination indicated improved thermal contact between the THKP and BW, hypothesized as a result from localized powder melting, although not confirmed. Increased thermal contact between BW and conductive powder lowers a device's resistance, leading to reduced power at constant current firing, and longer ignition times. Resistivity measurements on THKP pellets subjected to a 5 kV ESD discharge resulted in dramatic decreases in resistivity for lower density, highly hydrided pellets, which White [4] theorized may arise from fractures in the oxide coating.

Investigations into the mechanism causing the significant resistance drop (up to 33%) was shown [1] to be isolated to the TKP powder, because

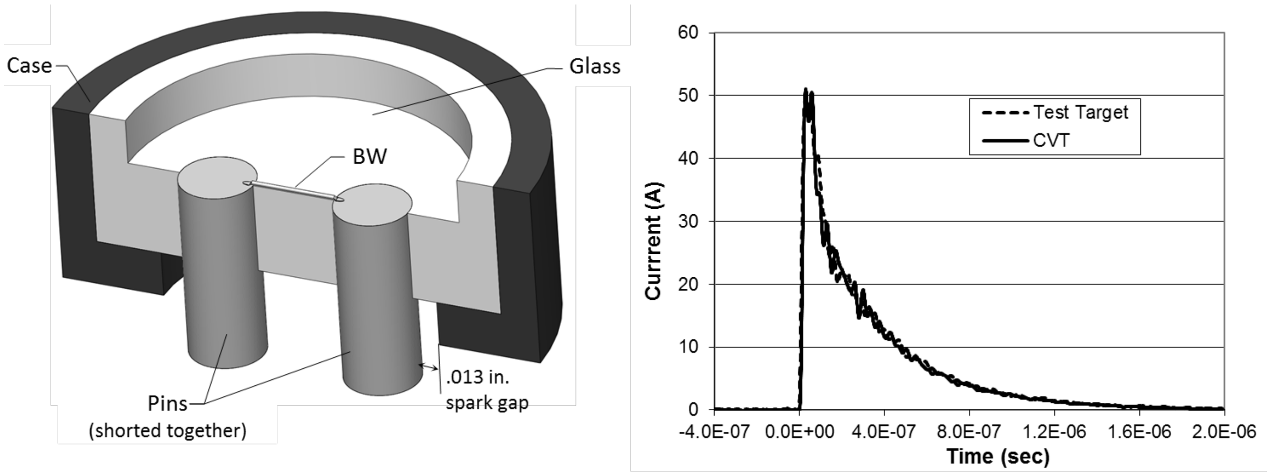
- unloaded, bridged headers showed no change in resistance after tens of ESD pulses, ruling out degradation to the header body (e.g. parallel path developing in the spark gap), and
- ESD-degraded ignitors, whose TKP was subsequently removed, returned to their pre-loaded resistance, demonstrating no enduring effect to the header's BW-pin interface.

Two mechanisms were identified as causing the resistance change. First was the interaction with the BW overlapping the pins. Welding the BW on the pin-edges eliminated resistance changes that had been observed from ESD on ignitors loaded with inert (non-conductive) powder. Second, the effect was further isolated to the TKP when ignitors built without a BW were subjected to repeated ESD pulses and their resistance changed from an open circuit to a resistance of 3-4 Ω as a parallel path developed through the TKP. However, no visual change (i.e. burning) was apparent to the TKP when viewed under microscopy or with Scanning Electron Microscope (SEM).

TKP is a pyrotechnic that has seen widespread use in ignitors for 80 years [5]. This work continues to study how TKP behaves in a standard ignitor design when subjected to ESD insults. TKP with differing mix ratios, densities, powder geometries, titanium particle size distributions, and morphologies were subjected to increasing ESD discharge voltages and repeated pulses.

2 Experimental Setup and Procedure

The ignitor header used during testing consisted of two pins electrically isolated from each other and the stainless steel 304L header shell by a Corning equivalent sealing glass and had a 0.33 mm (0.013 in.) air (spark) gap designed to breakdown between the pins and shell at approximately 2200 V. Ignitor geometry (Figure 1a) consisted of 1 mm (0.04 in.) diameter pins with the BW resistance-welded on pin centerline resulting in a nominal resistance of 1.1 Ω prior to loading the pyrotechnic. Care was taken to weld the BW at the edges of the pins.



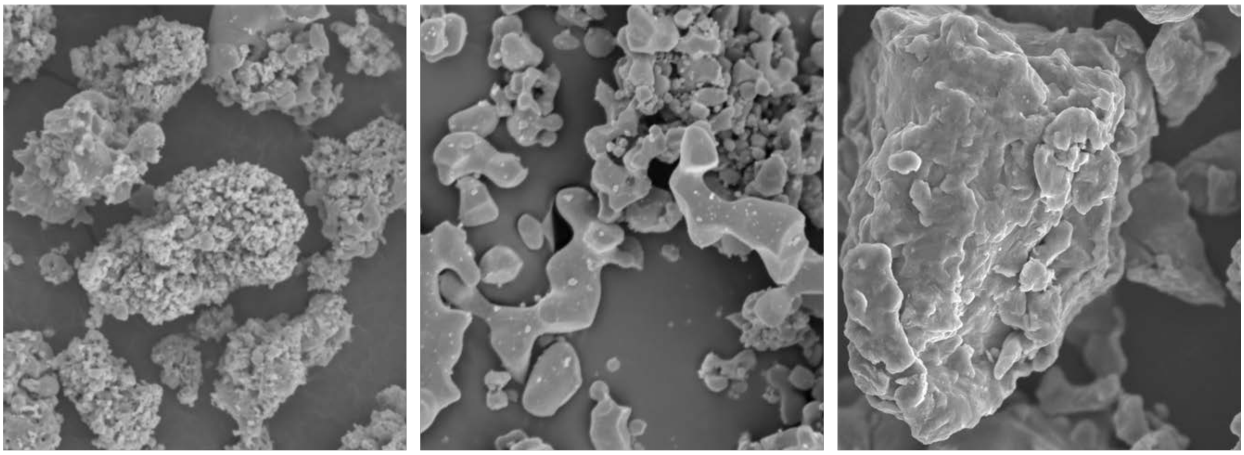
(a) Cross section of header geometry (b) ESD 25 kV discharge waveform

Figure 1: Schematic of ignitor header and 25kV discharge ESD waveform delivered to ignitor.

Ignitors were loaded with either titanium potassium perchlorate (TKP), boron calcium chromate (BCC), or a mixture of BCC and TKP (BCTK) by pressing the powder directly on the pins and BW to the desired pressure (34, 69, 103, or 138 MPa). The applied force to the compacted powder was measured by a load cell at the base of the header, and converted to pressure using the pressing ram cross sectional area. TKP-I powders had a 0.33/0.67 by weight mix ratio of Ti metal and potassium perchlorate ($KClO_4$) powders. TKP-II had a 0.41/0.59 mix ratio. Four TKP powders were tested, which had particle size distributions 13-21 µm (50th percentile), and whose Ti particles differed in surface area:

- TKP-Ia – surface area <15 m^2/g
- TKP-Ib – ~1/2 the surface area of TKP-Ia
- TKP-Ic – ~1/4 the surface area of TKP-Ia
- TKP-II – ~1/40 the surface area of TKP-Ia

The titanium particles in TKP-Ia and TKP-Ib and similar morphologies (Figure 2), although they differed in surface area. BCTK was a 0.5/0.5 mixture of BCC and TKP-Ic. $KClO_4$ had a 50th percentile particle size 9-22 µm, depending on the TKP.



(a) TKP-Ia and TKP-Ib (b) TKP-Ic (c) TKP-II

Figure 2: Morphologies of titanium powders in TKPs tested.

Compacted powder density was calculated from the header cavity diameter and pressed powder height and mass. Although pyrotechnic types were pressed to the same forces (pressures), variance in powder density at the same pressing force arose as a result of friction from differing particle size distributions and surface areas effecting powder compaction. Frictional interactions between particles and with the cavity walls would also result in density gradients across the pellet height with larger density gradients expected for increasing heights. No estimation was made of the density gradient through the compacted powder.

Resistance was measured pin-to-pin across the BW using a calibrated Valhalla Scientific 4314 Ignitor Tester during ignitor assembly following bridging and again after powder pressing for the completed ignitor. Resistance was again measured prior to ESD testing and following each ESD pulse.

The Electro-Tech Systems (ETS) Model 931 Firing Test System provided the ESD waveform, which was consistent with MIL-STD-331C, having a risetime of ~15 ns. MIL-STD-331C requires a 500 Ω resistance, 500 pF capacitance, and <5 µH inductance. Current waveforms were recorded before each test series by discharging the ESD pulse into the 2-Ω ETS Model 949 Test Target, as well as monitored while testing the ignitors with a Pearson Model 2877 current viewing transformer (CVT). The CVT had bandwidth extending in to the GHz range (2 ns risetimes). An example ringdown for a 25 kV discharge is shown in Figure 1b. Good agreement is shown between the Test Target and the Pearson CVT. The ESD waveforms shown in Figure 1 are double exponential fits to the recorded data as recommended by JOTP-062 [6].

The energy delivered to the ignitor (the area under the square of the current) was calculated for the ESD ringdown waveform and verified that it matched the 0.18-0.22% allowable range designated by MIL-STD-331C. The ESD pulse was typically applied pin-to-case, with one electrode attached to the two pins (shorted together) and the other electrode to the ignitor case.

3 Results and Discussion

Groups of ignitors were built with differing pyrotechnic type pressed at 34, 69, 103, or 138 MPa on the BW of the standard header and exposed to ESD. From each group, a minimum of four ignitors received at least fifteen ESD pulses at 25 kV. Ignitors from each group were exposed to ESD with increasing discharge voltages (2, 3, 4, 5, 10, 15, 20 and 25 kV) applied pin-to-pin and pin-to-case. Table 1 compares the pyrotechnic loading parameters and results for the different groups. The pyrotechnic loading resistance drop is the decrease in resistance following powder compaction on the BW compared the bare BW resistance. The ESD resistance drop is the decrease in resistance (after a given number of ESD pulses) compared to the pre-ESD resistance.

Table 1: Ignitors Exposed to Repeated 25 kV ESD Pulses Pin-to-Case.

					Average Resistance Drop (%)			
Pyro Type	# Tested at 25 kV	TKP Mass (mg)	Pressing Pressure (MPa)	Density (g/cc)	Pyro Loading	1 ESD pulse	5 ESD pulses	≤15 ESD pulses
BCC	4	30	103	1.58	0.0%		0%	
BCTK	4	30	69	1.93	0.1%			
	4	30	103	2.00	0.0%		0%	
	4	30	138	2.08	0.2%			
TKP-Ia	5	30	34	1.63	0.4%	4.7%	7.7%	8.8%
	5	30	69	1.90	6.6%	8.9%	10.8%	12.0%
	4	20	103	2.06	11.8%	8.3%	12.4%	12.5%
	4	25	103	2.04	11.1%	6.9%	11.3%	12.5%
	4	30	103	2.03	8.2%	6.0%	8.3%	11.1%
	180	30	103	2.02	12.6%	7.1%	10.8%	NA
	5*	30	103	2.06	13.4%	9.7%	11.8%	13.4%
	5	30	138	2.15	17.0%	8.8%	9.6%	13.9%
TKP-Ib	4	30	69	1.91	7.8%	5.8%	6.9%	9.0%
	4	30	103	2.04	15.2%	3.6%	6.4%	8.7%
	4	30	138	2.15	20.5%	5.9%	9.1%	8.0%
TKP-Ic	4	30	69	2.17	0.1%	3.5%	9.6%	10.2%
	4	30	103	2.31	0.1%	5.6%	15.2%	17.3%
	4	30	138	2.39	0.3%	12.0%	18.9%	19.1%
TKP-II	5	20	103	2.45	4.3%	21.0%	25.8%	27.3%
	5	30	103	2.45	7.3%	19.8%	25.6%	27.7%

*BW raised off glass header

Ignitors loaded with BCC and BCTK showed no change in resistance across all pressing pressures either following pyrotechnic loading on the BW or after repeated ESD insults. Boron has a high electrical resistivity (~10^6 Ω-m) compared to the 0.01-1 Ω-m resistivity of TKP [7].

Resistances for TKP loaded ignitors decreased for increasing powder pressing pressures for all TKP types except TKP-Ic, which showed virtually no change in resistance regardless of compacted powder density. The metallic TKP when pressed onto the BW provides a parallel conductive path, so that the equivalent resistance is less than the resistance measured through the BW

$$R_{eq} = \frac{R_{BW}R_{TKP}}{R_{BW}+R_{TKP}} \tag{1}$$

TKP-Ic and TKP-II had higher densities at the sample pressing pressures, indicating reduced friction between particles and the header cavity walls. Even though TKP-II had a higher proportion of Ti in TKP, it had a substantially lower resistance drop from loading compared to TKP-Ia and TKP-Ib, implying lower metal to metal contact throughout the compacted powder or higher electrical contact resistance between Ti particles.

For TKP ignitors, resistance decreased with the increasing number of ESD pulses, regardless of test group. Roughly 80% of the resistance change, from 15 pulses, occurred within the first 3-4 pulses for all TKP types as shown in Figure 3 by the cumulative resistance change as a function of ESD pulses applied at 25 kV. Between six and fifteen pulses the resistance changed only minimally, have reached ~90% or better of the total resistance change. Ignitors tested to 100 ESD pulses only changed by an additional 10% between 30 and 100 pulses.

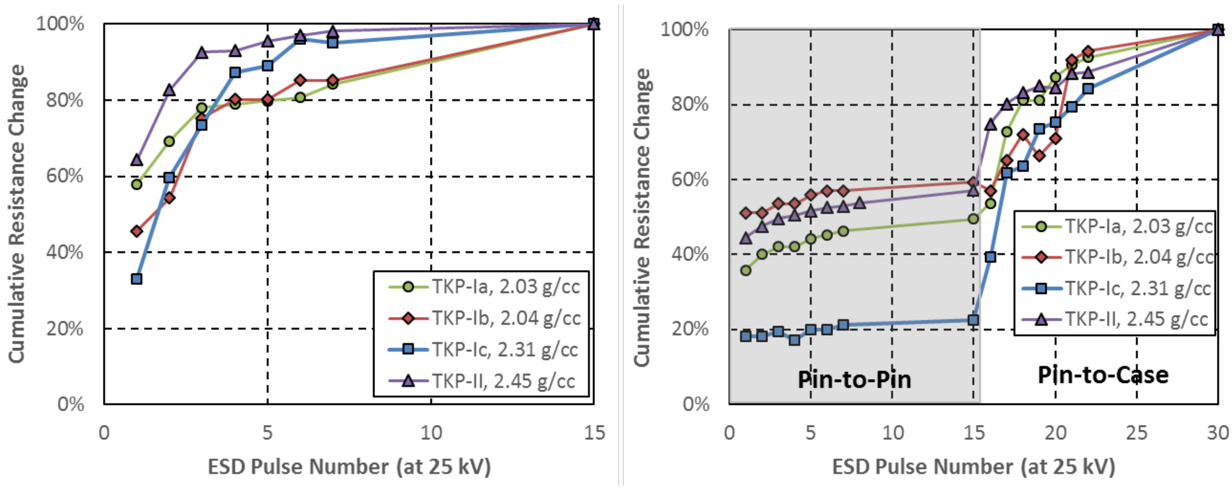


Figure 3: Cumulative resistance change as a function of the number of ESD pulses applied at 25 kV (left). Cumulative resistance change for 25 kV ESD applied pin-to-pin and then pin-to-case (right).

For each TKP type, fifteen 25 kV ESD pulses were applied pin-to-pin, followed by fifteen pulses pin-to-case. Pin-to-pin resistance change accounted for 20-60% of the cumulative resistance, depending on TKP type (Figure 3) with TKP-Ic showing the least change in resistance. Pressing density had only minimal effect, although as expected the lowest density showed the least resistance change for all TKP-I (Figure 4). The ESD resistance drop for ignitors subjected to pin-to-pin and pin-to-case (30 pulses total) was similar to other test groups pressed at the same pressure that received fifteen 25 kV ESD pulses pin-to-case (*e.g.* 8.5% for TKP-Ib 2.04 g/cc compared to 8.0%).

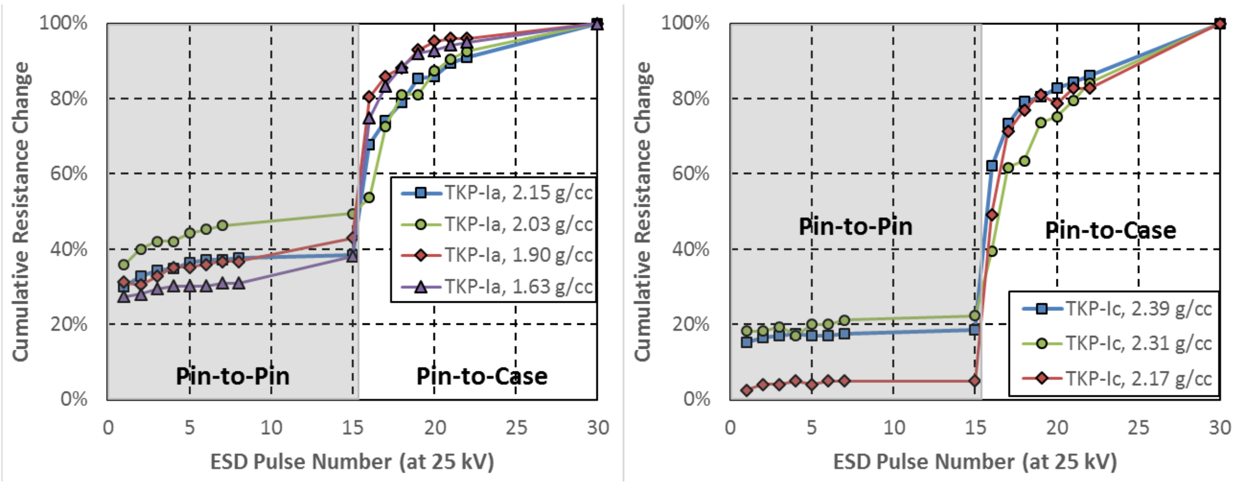


Figure 4: Cumulative resistance change for 25 kV ESD applied pin-to-pin then pin-to-case for different densities of TKP-Ia (left) and TKP-Ic (right).

Variability was observed within most test groups, likely as a result of compacted powder microstructure (e.g. titanium metal contact, voids, and powder bed density around BW). For one group of TKP-Ia ignitors, the BW was raised off the header glass by bridging over a 5-mil wire spacer, so that TKP could pack more readily around the BW. ESD testing was also performed on another much larger test group (180 ignitors) to compare results against the smaller test groups. Results for the raised BW group and larger sample group showed good agreement with other TKP-Ia ignitors pressed at 103 MPa, regardless of TKP mass.

The effect of conductive powder depth was investigated to determine the depth to which ESD was affecting (penetrating) the compacted TKP. A thinner TKP height should show less resistance change from ESD (and powder loading). Ignitors were built with 20, 25, and 30 mg of TKP-Ia pressed at 103 MPa, resulting in TKP heights ~20-30 times the BW diameter. Density increased slightly with decreasing TKP mass (height). Resistance changes from loading TKP increased with increasing density (Table 1), as it did following ESD (Figure 5). Further investigation is required at even lower TKP masses (heights) to determine if there is a measurable critical height, at which ESD has a reduced effect of resistance change.

Ignitors from each test group were subjected to ESD pulses with increasing discharge voltages (2, 3, 4, 5, 10, 15, 20, and 25 kV). Little change occurred until 3-4 kV. Resistance changes increased significantly between 3 and 5 kV with TKPs ignitors seeing 30-60% of the cumulative resistance change by 5 kV. Resistance changed approximately linearly between 5 and 25 kV (Figure 5).

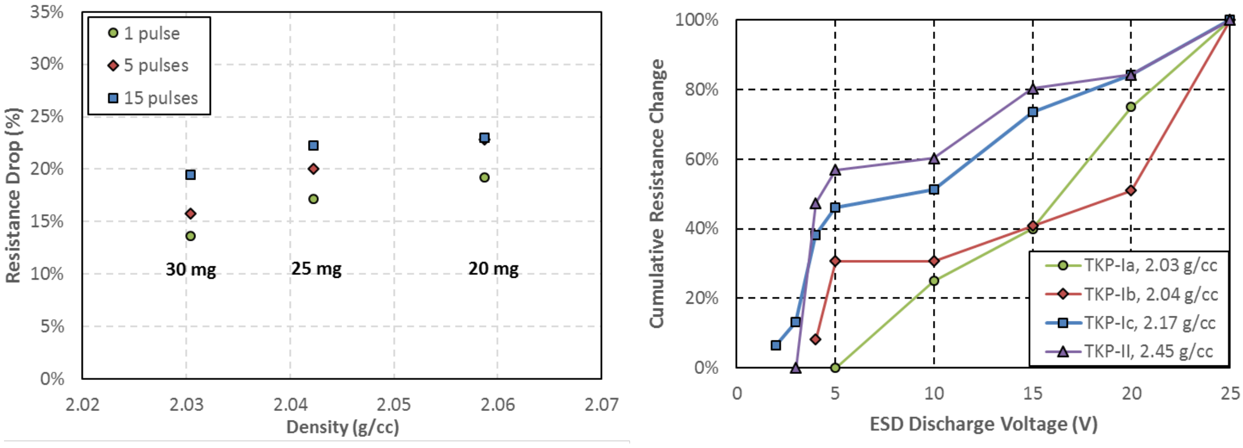


Figure 5: Effect of TKP height (mass) on resistance change (left). Example of cumulative resistance change for ESD pulses with increasing discharge voltage (right).

The effect of pyrotechnic type and density on resistance change following ESD became clearest when the ignitor's total resistance change (from TKP loading and ESD) was considered. As discussed previously, lower resistances resulted from TKP pressed at higher densities. Figure 6 shows the resistance change following one and fifteen ESD pulse(s) as a function of TKPs' theoretical maximum density ($TMD_{TKP\text{-}I}$ = 2.955, $TMD_{TKP\text{-}II}$ = 3.07 g/cc). Comparisons indicate that TKP-Ia and TKP-Ib, which shared common Ti morphologies, particle sizes, and compaction densities although with different Ti surface areas, behaved similarly. In contrast, TKP-II and particularly TKP-Ic showed lower change in resistance for the same powder density. TKP-Ic and TKP-II pack to higher densities at the same pressing pressure. TKP-Ia and TKP-Ib also compared well against previous ESD tests [1], which used a larger ignitor (labeled G2) loaded with 40 mg TKP-Ia pressed on the BW (with 110 mg TKP-II on top). The larger G2 ignitor had a substantially lower TKP-Ia height (less than 13x BW diameter). Results after fifteen ESD pulses (9 pulses for G2 ignitors) showed the best agreement (Figure 6, right).

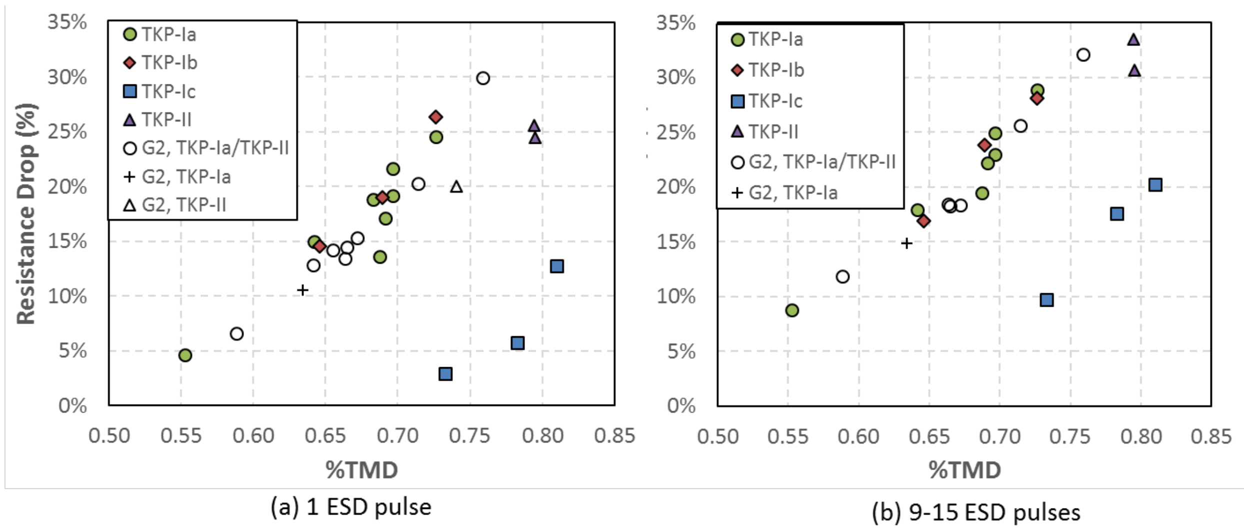


Figure 6: Resistance drop following a single ESD pulse (left) and 9-15 ESD pulses (right) for TKP-loaded ignitors in various test groups.

4 Conclusions

Significant changes in ignitor resistance occurred following application of ESD to ignitors that include a spark gap built into the headers to protect against ESD and prevent inadvertent ignition. It is not well understood how ESD produces such effects on ignitors with features designed to protect the energetic powder.

Increasing TKP density resulted in higher resistance decreases with total resistance change from TKP loading and ESD insult(s) providing the best correlation. TKP types that differed in particle size distribution and Ti particle morphology resulted in different magnitudes of change in resistance, although Ti particle surface area appeared to have minimal effect. TKP-Ia and Ib, which shared many similarities except Ti surface area, behaved comparably, whereas TKP-Ic showed significantly lower resistance changes at the same density. TKP-II, which had a higher Ti to $KClO_4$ mix ratio than TKP-I, counterintuitively demonstrated lower resistance change.

The change in resistance was not just a surface effect, but extended into the TKP pressed powder pellet, although the depth of the ESD effect remains to be determined.

For all but one case (TKP-Ib, 2.04 g/cc), pin-to-case ESD resulted in higher resistance change than pin-to-pin. Significantly higher for TKP-Ic, regardless of density. Although typically no changes in resistance occurred less than 3 kV, ignitor resistance decreased significantly between 3-5 kV and continued almost linearly through 25 kV (the highest discharge voltage tested). The majority of the resistance change occurs within the first five ESD insults, although minimal changes were still witnessed out to 100 insults.

Although macroscopic effects (resistance changes) following ESD for differing TKP types with varying densities and morphologies are better understood leading to techniques to reduce ignitor degradation, attempts are underway to pinpoint the root mechanism for change. Non-invasive micro-computed tomography (microCT) are underway in the hope to gain a better understanding of the state of the TKP and changes occurring particularly at the Ti to Ti particle interfaces.

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6 References

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