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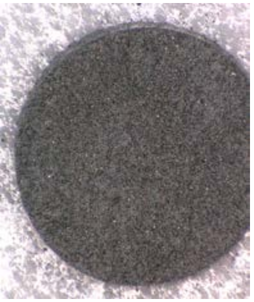

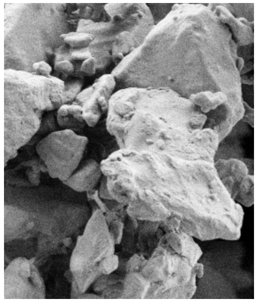

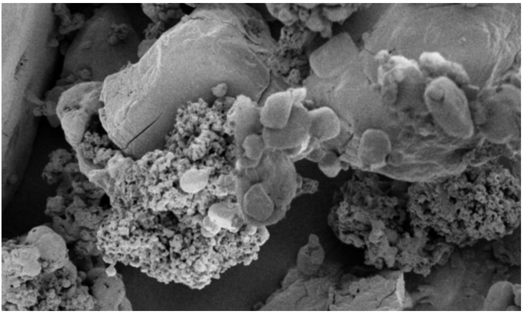

Coefficient of Thermal Expansion of Titanium Potassium Perchlorate

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Exceptional service in the national interest

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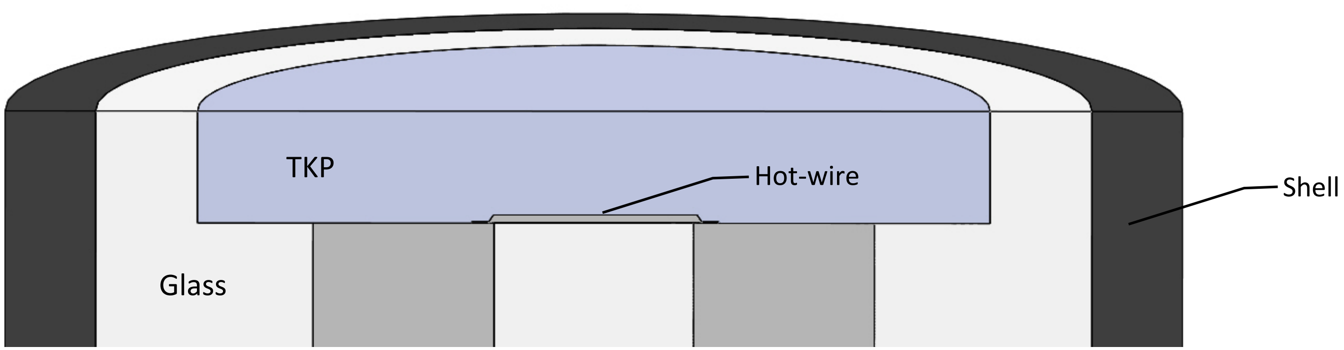
Overview

- Motivation
- Pyrotechnic tested
  - Titanium potassium perchlorate (TKP) variations
- Sample preparation – compacted powders
  - Varied density – pressed to force
  - Pressing temperature
- Instrumentation and test methodology
  - Thermomechanical Analyzer (TMA)
  - Temperature range -65 to 95 ^{o}C
- CTE comparison by TKP type
- Conclusions

Motivation

- Reliable ignitor functionality – influenced by intimate contact between pyrotechnic and hot-wire
- Ignitor materials must expand/contract in unison during thermal exposure
- Pyrotechnics with substantially higher CTE – more dramatic dimensional change
- Contraction of pyrotechnic can lead to decoupling and reduced thermal conduction from hot-wire, affecting performance
- CTE measurements could be used to assist modeling predictions

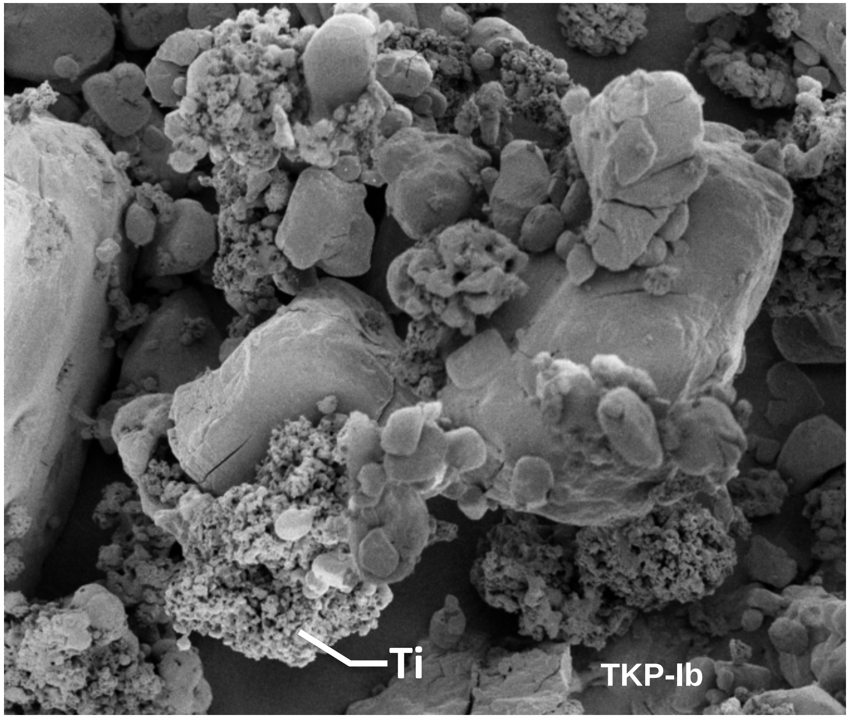


Ignitor Schematic

Pyrotechnic Materials

- Four TKP powders tested – differed in:
  - Ti/$KClO_4$ mix ratio
  - Particle size distribution (PSD) – 13 < Dia_{50th}[μm] < 21
  - Surface Area (SA) – 1/40
  - Morphology - titanium

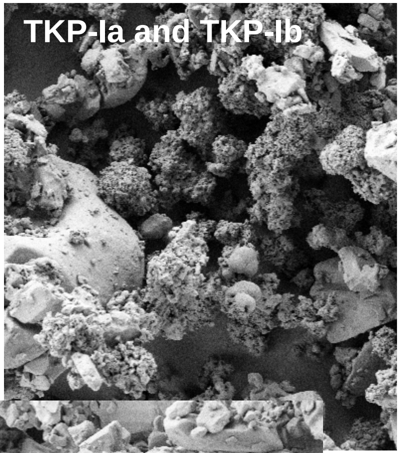
- Titanium subhydride potassium perchlorate ($TH_{1.65}KP$)
  - Evaluated for comparison to literature
- $KClO_4$ also evaluated

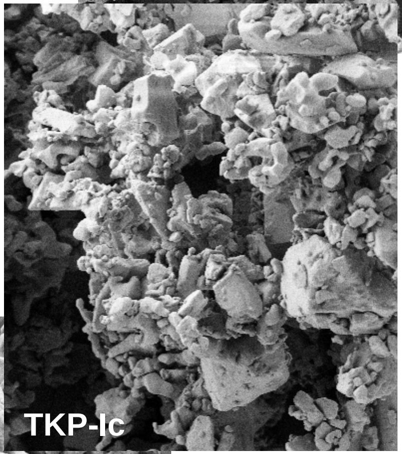


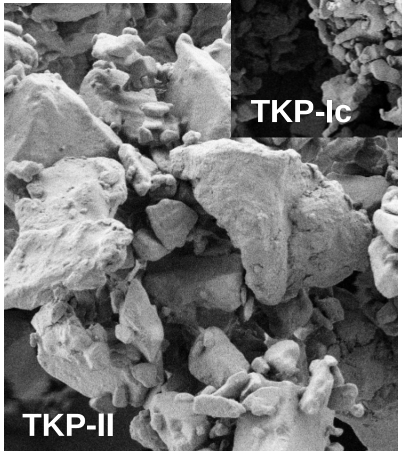
Titanium Potassium Perchlorate

- TKP-I and TKP-II differed in Ti/$KClO_4$ mix ratio
- TKP-Ib – similar morphology, Ti PSD as TKP-Ia
  - ½ surface area of TKP-Ia
- TKP-Ic - different morphology
  - ¼ surface area TKP-Ia
- TKP-II – titanium with 1/40 SA TKP-Ia
- TKP-Ic and TKP-II packed denser at same force

Material	Mix Ratio Ti/$KClO_4$	50th Percentile Particle Size (µm) Ti	$KClO_4$	Surface Area (m^2/g) Ti	$KClO_4$
TKP-Ia	0.33/0.67	$13<D_{Ti}<21$	$D_{KP}<10$	$SA_{Ti}<15$	$SA_{KP}<1$
TKP-Ib	0.33/0.67	D_{Ti}	$\sim(2*D_{KP})$	$\sim SA_{Ti}/2$	$\sim SA_{KP}/2$
TKP-Ic	0.33/0.67	D_{Ti}	D_{KP}	$\sim SA_{Ti}/4$	SA_{KP}
TKP-II	0.41/0.59	D_{Ti}	D_{KP}	$\sim SA_{Ti}/40$	SA_{KP}
$KClO_4$	0/1	—	$\sim(2*D_{KP})$	NA	$\sim SA_{KP}/2$
$TH_{1.65}KP$	0.33/0.67	<10	$\sim D_{KP}$	$\sim SA_{Ti}/5$	SA_{KP}



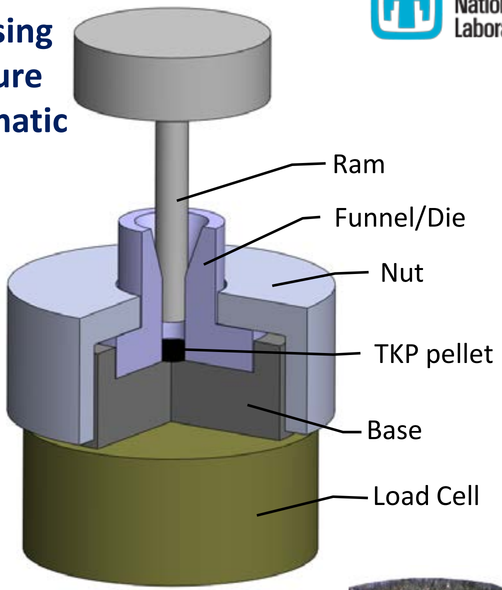




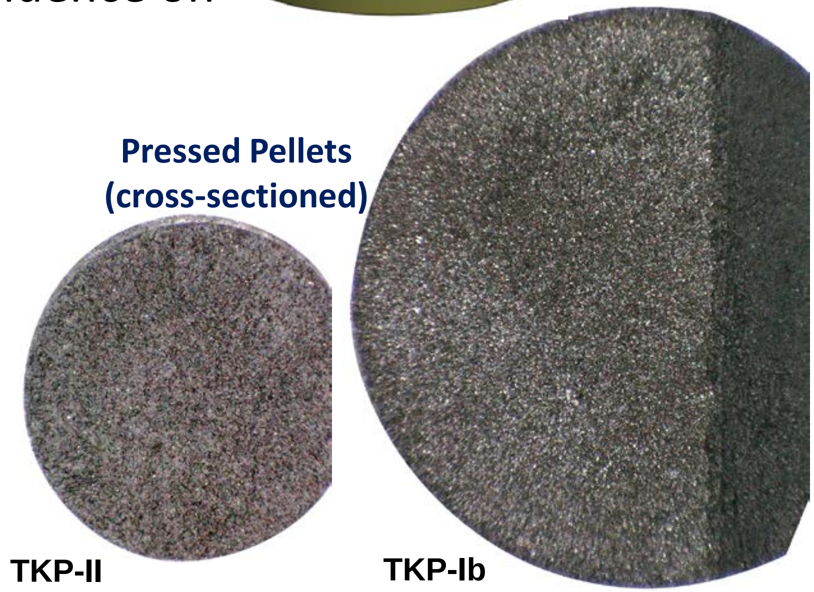
Sample Preparation

- TKP powder compacted by pneumatic press to desired pressure
  - 69 or 103 MPa – densities (1.9-2.41 g/cc) differed depending on TKP type
- Powder mass and diameter initially varied to evaluate pellet friability and geometric influence on CTE
  - Mass – 106-319 mg
  - 3.8 or 6.4 mm cylindrical pellets
  - Length/Diameter – 0.43-1.07
- Pressing temperature
  - Ambient
  - 85 ^{o}C

Pressing Fixture Schematic



Pressed Pellets (cross-sectioned)

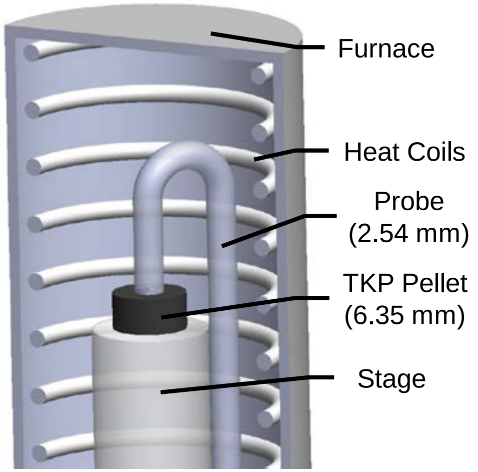


Experimental Setup

- TA Instruments Thermomechanical Analyzer
  - Measured axial dimension changes
  - Temperature range -65 to 95 ^{o}C, 3 ^{o}C/min ramp
  - Helium purge gas (100 mL/min)
    - Furnace temperature uniformity – heating coils and LN cooling head
  - Expansion probe – preloaded force (0.1 or 0.2 N)
    - Ensured no sample movement, bias, or damage

TA Instruments Q400 Thermomechanical Analyzer (TMA)

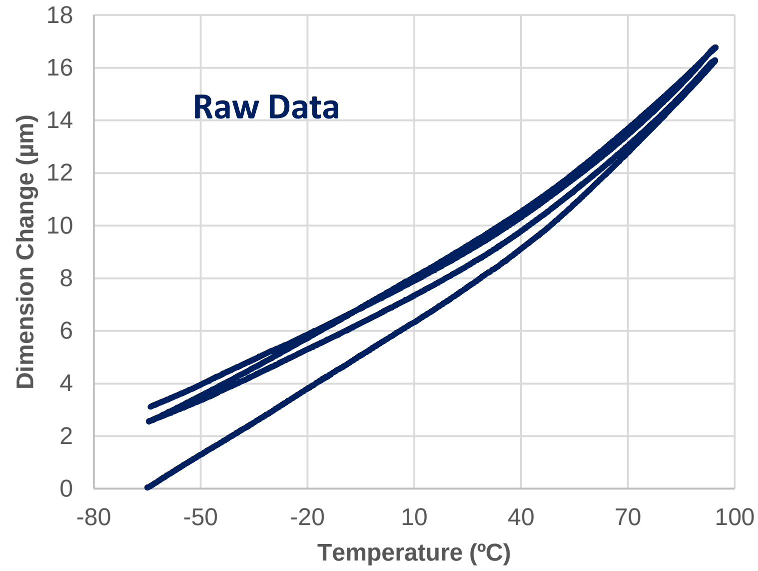
TMA Furnace Schematic

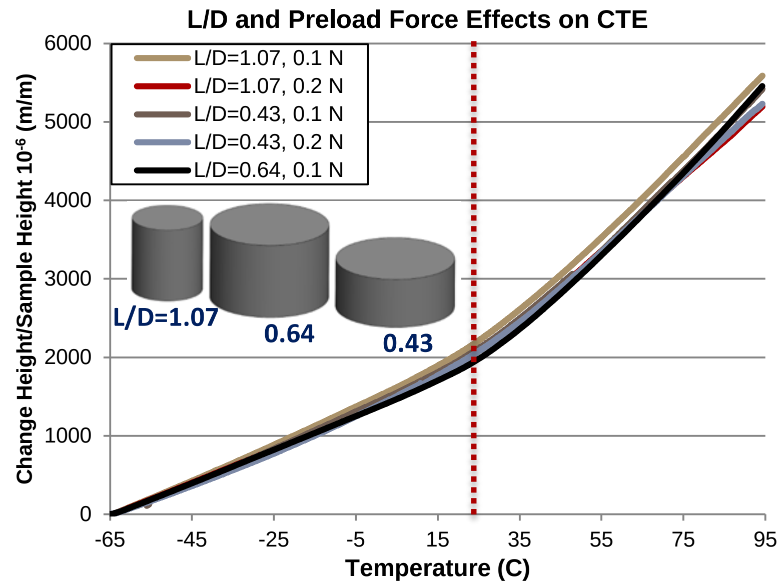


- Calibration
  - Force, temperature, and probe
- Aluminum standard used to calibrate CTE

Test Methodology and Refinement

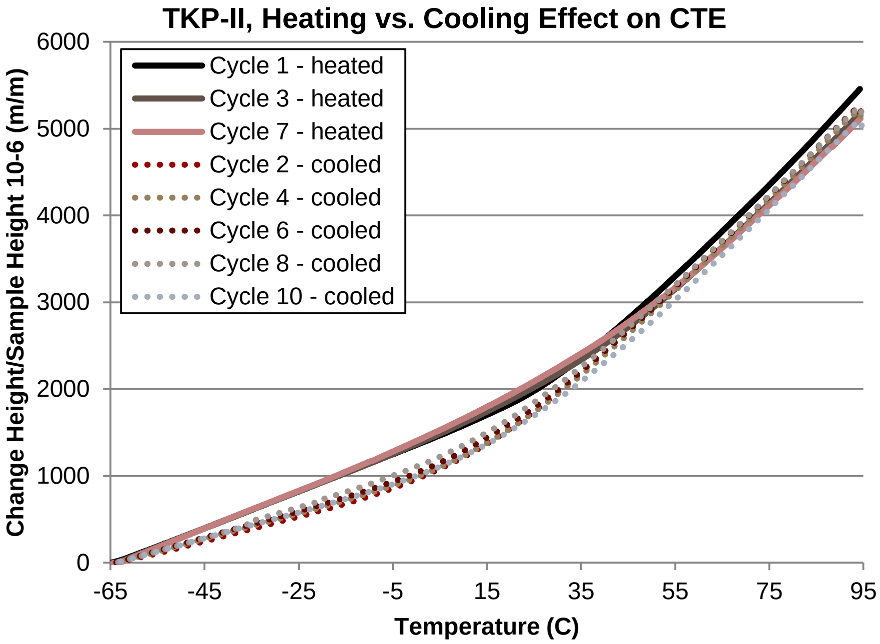
- TKP-II pellets – refined TMA settings and pellet geometry, varying:
  - Length/Diameter – 0.43, 0.64, 1.07
  - Probe preload force – 0.1 or 0.2 N
- CTE – slope of height change (per pellet thickness) versus temperature
- Good agreement across L/D and preload forces
- 3.8 mm diameter pellets – susceptible to damage
- Subsequent samples used:
  - L/D ≈ 0.5 (~200 mg)
  - 0.1 N preload force
- Slope change between 20-30 ºC





Heating versus Cooling Cycles

- CTE profile during heating cycle differed from the cooling cycle (hysteresis), for most samples
- CTE from initial heating cycle slightly higher to other heating cycles
  - Indicates slight irreversible change in compacted powder
- Subsequent heating cycles agreed (cooling cycles agreed)
  - 10 cycles studied
- Subsequent samples – 4-6 cycles
- Comparisons made for Cycle 3 (heating)



Results – CTE Measurements

- CTE for $TK_{1.65}KP$ comparable (slightly higher) to $TK_{0.65}KP$ [Massis]
- CTE reported for three temperature ranges
- CTE slope change
  - CTE increased with temperature
  - Occurs at about room temperature
  - Influenced by pellet pressing temperature?

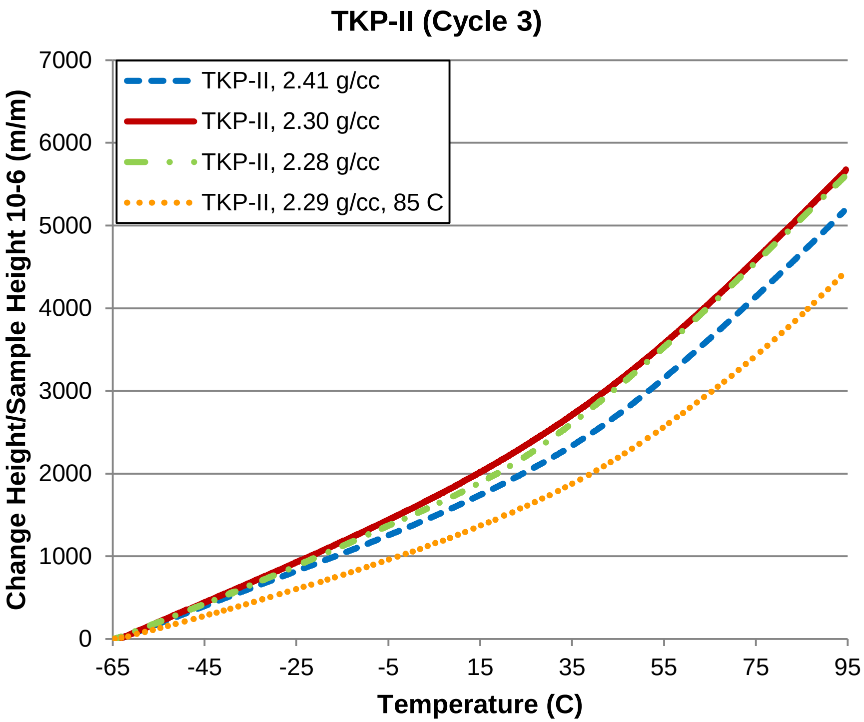
Pyrotechnic	CTE (x10^{-6}/ºC)		
	-50 to 0	0 to 50	50 to 95 ºC
$TH_{1.65}KP$ (2.1 g/cc)	40	46	54
$TH_{0.65}KP$ [Massis]	33	42	50

Material	Pressing Temp	L/D	Density (g/cc)	CTE (x10^{-6}/ºC)			Hysteresis
				-65 to 20	20 to 95	-65 to 95 ºC	
TKP-II	Ambient	0.43-0.64	2.41	20	47	33	Y
TKP-II	Ambient	0.45	2.30	24	50	36	Y
TKP-II	Ambient	1.07	2.28	23	47	34	Y
TKP-II	85 °C	0.45	2.29	15	44	28	Y
$TH_{1.65}KP$	Ambient	0.46	2.23	39	50	44	N
$TH_{1.65}KP$	Ambient	0.49	2.10	40	52	46	N
TKP-Ia	Ambient	0.49	2.05	26	43	34	Y
TKP-Ia	Ambient	0.52	1.91	25	39	32	Y
TKP-Ib	Ambient	0.49	2.05	21	33	26	Y
TKP-Ib	Ambient	0.53	1.87	23	36	29	Y
TKP-Ic	Ambient	0.45	2.27	22	43	32	Y
TKP-Ic	Ambient	0.49	2.05	32	47	40	NA
$KClO_4$	Ambient	0.45	2.20	63	73	68	N
$KClO_4$	Ambient	0.49	1.98	61	73	66	N

Massis, T.M.; Morenus, P.K.; and Steele, B.R., The Simulation of Powder-Decoupling Phenomena in Electro-Explosive Devices by Thermomechanical Analysis (TMA), presented at the Joint Symp. on Compatibility of Plastics and Other Matter with Explosives, Phoenix, AZ., May 30, 1982.

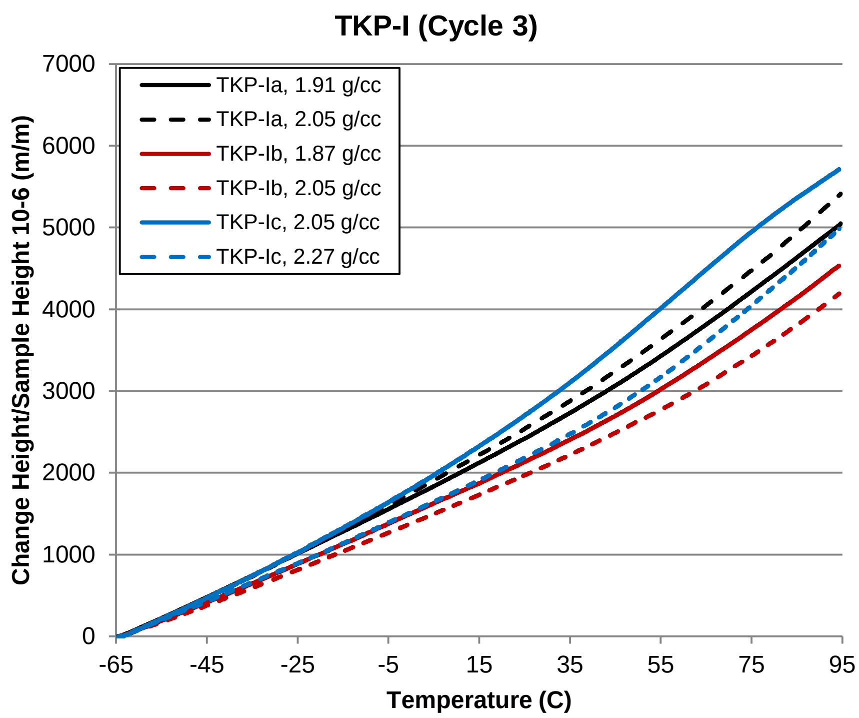
TKP-II Results

- Data repeatable between samples
- Slight density dependence – lower density, typically slightly higher CTE
- Pellet pressed at 85 ^{o}C – attempt to influence CTE
- Result had noticeable shift in CTE (lower)



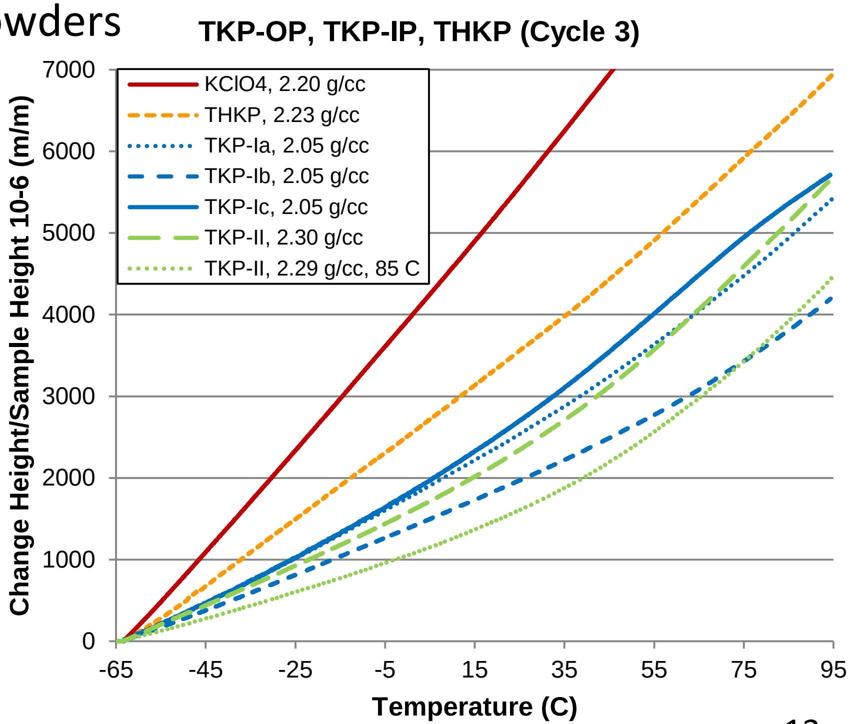
TKP-I Results

- TKP-I had wider spread in CTE than TKP-II
  - Wider range of powder characteristics (morphology, PSD, etc.)
- Lower densities typically had slightly higher CTE



Pyrotechnic Comparison

- Compared samples pressed at 103 MPa
- TKP-I and TKP-II comparable
- TKP lower than $TK_{1.65}KP$
- $KClO_4$ tested for comparison
  - Driver for higher CTEs in compacted powders



Conclusions

- Powder Ti/$KClO_4$ mix ratio, morphology and compaction density had minimal effect on CTE
- Initial heating cycle had highest CTE
  - Repeatable CTE for subsequent hot-cold cycling
- Dimension changes from heating and cooling demonstrated hysteresis
- Pressing powder at 85 ^{o}C (instead of ambient) resulted in CTE
- CTE for TKP consistently lower than THKP

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