

# Improving Oxidation of the Extrusion Melt at Higher Line Speeds and Lower Melt Temperatures

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# Introduction

The Time in the Air Gap (TIAG) for the last 20+ years has been recommended from 80 msec to 120 msec as the necessary exposure of the melt curtain to be properly oxidized and give adequate bonding to the substrate.

When this work was reported the extrusion coating and extrusion lamination line speeds were significantly slower than today's equipment.

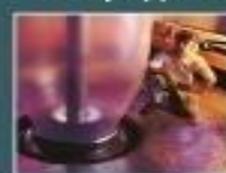
In order to achieve the same level of oxidation at these higher line speeds the reaction needs to be enhanced.



# Introduction

This presentation will show work done using ozone exposure at higher line speeds and lower melt temperatures to achieve and exceed the traditional TIAG recommended range.

In addition, by lowering the melt temperature and having the same level of oxidation to the melt curtain, the heat seal integrity can be improved.



# Introduction

The two equations used in the Industry to calculate the TIAG;

Du Pont Technical Presentation;

V. Antonov and A. Soutar, “*Foil Adhesion With Copolymers: Time in the Air Gap,*”  
TAPPI 1991 PLC Conference Proceedings, pp 553-574

Dow Chemical Company Microsoft Excel Spreadsheet;

Time in the Air Gap Calculator, Developed by Mark Heard of Dow Chemical Co.



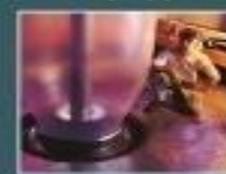
*Antonov and Soutar Air Gap Equation:*  
Calculating the Minimum and Maximum Line Speeds,  
using the maximum reasonable 25.4 cm (10 inch) Air Gap

$$\text{Time in the Air Gap (msec)} = \frac{[\text{Distance (mm)}] [60]}{[\text{Line Speed (mpm)}]}$$

$$\text{Line Speed (mpm)} = \frac{[\text{Distance (mm)}] [60]}{[\text{Time in the Air Gap (msec)}]}$$

$$\text{Line Speed (Min TIAG)} = \frac{[254 \text{ (mm)}] [60]}{[80 \text{ (msec)}]} = 191 \text{ mpm (626 fpm)}$$

$$\text{Line Speed (Max TIAG)} = \frac{[254 \text{ (mm)}] [60]}{[120 \text{ (msec)}]} = 127 \text{ mpm (413 fpm)}$$



## Time in the Air Gap – “Heard” Calculator

### *Parameters to input*

Resin mass flow rate (lb/hr)	1000
Resin density (g/cc)	0.924
Die width (in.)	110
Die gap (mils)	25
Line speed (ft/min)	1000
Air gap (in)	10

### *Calculated values*

Resin velocity at die (ft/min)	15.1
Average velocity (ft/s)	507.6
Time in the air gap (milliseconds)	98.5

### Metric units

454 (kg/hr)
0,924 (g/cc)
279 (cm)
0,635 (mm)
305 (m/min)
254 (mm)
4,6 (m/min)
155 (m/sec)
98,5 (msec)

A good rule of thumb:

Add 25,4 mm (1“) of Air Gap for every 30,5 mpm (100 fpm) of line speed.



## Equations for the “Time in the Air Gap – Dow Calculator”

$$\text{Resin Velocity at Die (fpm)} = \frac{[(\text{Resin Mass Flow Rate}) (454) (1000)]}{[(\text{Resin Density}) (\text{Die Width}) (\text{Die Gap}) (2.54)^3 (60) (12)]}$$

$$\text{Average Velocity (ft/sec)} = \frac{[(\text{Line Speed}) + (\text{Resin Velocity at Die})]}{(2)}$$

$$\text{TIAG (msec)} = \frac{[(\text{Line Speed}) (60) (\text{Air Gap})]}{[(12) (\text{Average Velocity})]}$$

$$\text{Air Gap (inches)} = \frac{[(\text{TIAG}) (12) (508)]}{[(1000) (60)]}$$

“Resin Mass Flow Rate” and “Velocity at Die” are the critical factors



## Air Gap Calculator

Min/ Max Line Speeds, using a 254 millimeter (10”) Air Gap

$$\text{Line Speed (Min TIAG)} = \frac{[ (\text{TIAG}) (12) (\text{Average Velocity}) ]}{[ (\text{Air Gap}) (60) ]}$$

$$\text{Line Speed (*Min* TIAG)} = \frac{[ (80 \text{ msec}) (12) (507.6 \text{ fps}) ]}{[ (254 \text{ mm}) (60) ]}$$

$$\text{Line Speed (Max TIAG)} = \frac{[ (\text{TIAG}) (12) (\text{Average Velocity}) ]}{[ (\text{Air Gap}) (60) ]}$$

$$\text{Line Speed (*Max* TIAG)} = \frac{[ (120 \text{ msec}) (12) (507.6 \text{ fps}) ]}{[ (254 \text{ mm}) (60) ]}$$



## Line Speed Limits for Recommended TIAG

$$\text{Line Speed (Min TIAG)} = \frac{[ (80 \text{ msec}) (12) (507.6 \text{ fps}) ]}{[ (254 \text{ mm}) (60) ]}$$

$$\text{Line Speed (Min TIAG)} = \mathbf{376 \text{ mpm} (1,234 \text{ fpm})}$$

$$\text{Line Speed (Max TIAG)} = \frac{[ (120 \text{ msec}) (12) (507.6 \text{ fps}) ]}{[ (254 \text{ mm}) (60) ]}$$

$$\text{Line Speed (Max TIAG)} = \mathbf{249 \text{ mpm} (817 \text{ fpm})}$$

These calculations are more accurate than the simpler equation.



## Line Speed Limits for Recommended TIAG

Using the Antonov and Souter Equation;

- min useable air gap - 178 mm (7")
- max useable air gap - 254 mm (10")
- range for line speeds between 134 to 191 mpm (440 to 625 fpm)

Using the Dow Chemical Company Equations;

- min useable air gap - 178 mm (7")
- max useable air gap - 254 mm (10")
- range for line speeds between 261 to 376 mpm (855 to 1234 fpm)

*Most Companies want to run the Extrusion Line between  
457 to 610 mpm (1500 fpm to 2000 fpm)*



## Recommended TIAG versus Line Speed

When comparing these line speed limits with today's equipment and companies wanting to run their lines between

460 - 610 mpm (1500 - 2000 fpm)

The air gap is not enough to give the proper oxidation to the melt.

Ozone blanketing, in addition to the Time in the Air Gap

is necessary to get proper oxidation in the melt.



# Experimental Parameters

To determine the limits of oxidation in these experiments, the following parameters were varied;

- Ozone exposure
- Melt temperature
- Air gap
- Line speed

Samples were run without ozone exposure, then the ozone was turned on and a second sample was exposed to ozone.

The experiments started with the highest melt temperature, normal extrusion coating conditions.

After the ozone exposed sample was collected, the melt temperature was reduced for the next sample.



# Experimental Parameters

- ✓ The melt curtain temperature was varied from normal extrusion melt conditions to very cold melt temperatures that without ozone would not give sufficient oxidation to the melt.
- ✓ The melt temperatures used were;

313°C (595°F)

299°C (570°F)

282°C (540°F)

304°C (580°F)

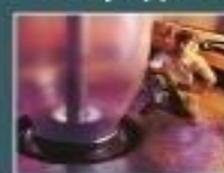
293°C (560°F)

277°C (530°F)

302°C (575°F)

288°C (550°F)

260°C (500°F)

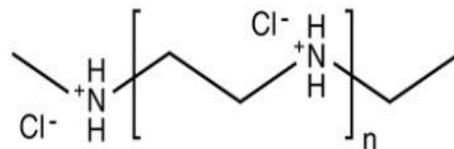


# Experimental Parameters

Two air gaps were used 178 mm (7") and 254 mm (10").

Two line speeds were used 183 mpm (600 fpm) and 366 mpm (1200 fpm).

A primer was applied to the film surface for all conditions, it was a modified Poly(ethyleneimine) primer.



Poly(ethyleneimine)

The polymer coat weight was held the same for all conditions; a proprietary resin blend was used, it is a polyethylene.



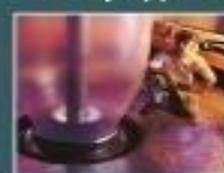
# Testing Conditions

The seal-ability and bond strength of the final structure was tested to determine the performance and indirectly the oxidation of the melt.

Bond performance evaluations were done by;

- “face to face” Heat Seals of the Sealant Layer
- Peel Tests (T-Peel)

*In order to do peel tests, a slip sheet was put through the extruder at each of the sample conditions.*



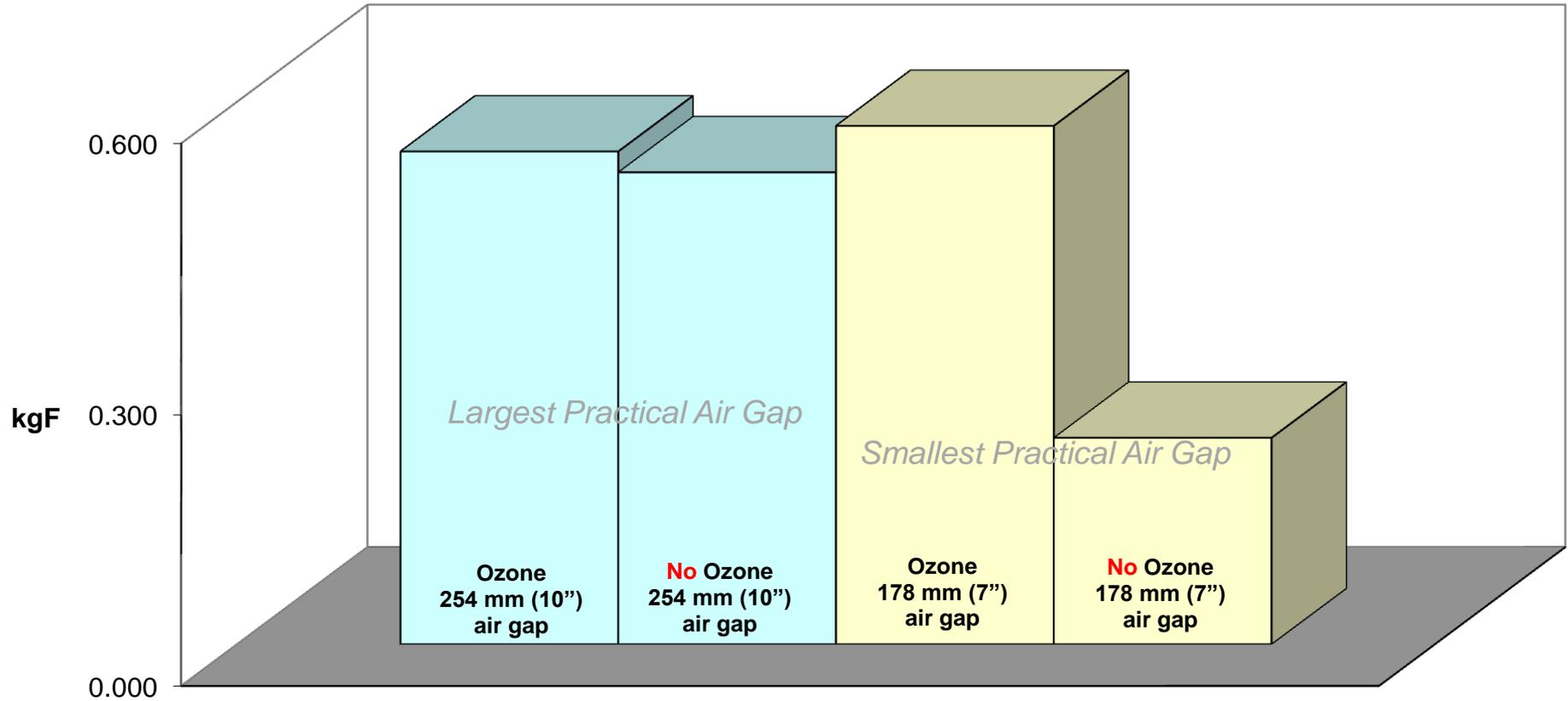
# Testing Conditions

- ✓ All samples were tested off-machine and then after several days of aging.
- ✓ The temperatures used for this presentation were;

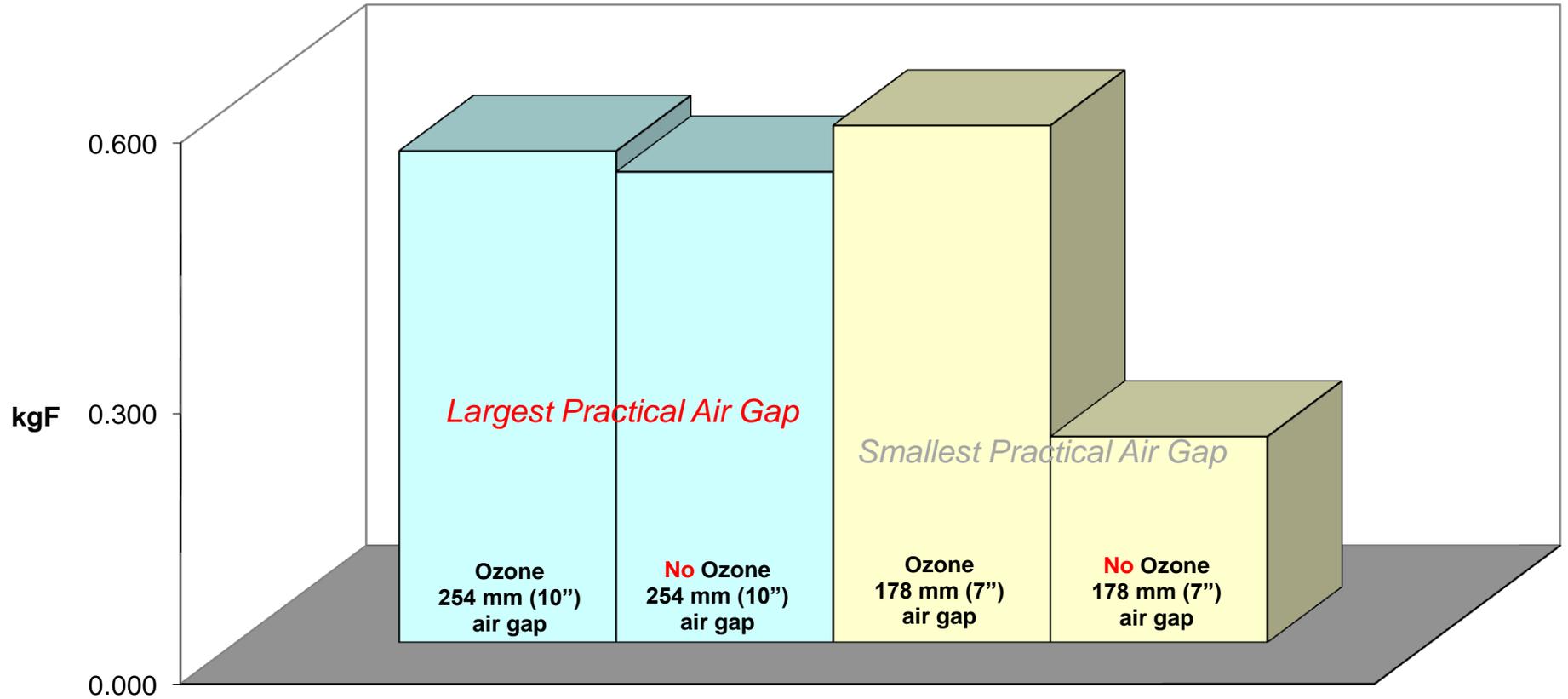
313°C (595°F)	282°C (540°F)
299°C (570°F)	277°C (530°F)
288°C (550°F)	262°C (510°F)
- ✓ Twenty separate samples were used to create the following graphs.
- ✓ This is a good cross-section that represents all of the data collected from this experiment.

Sample	Ozonator	Melt (F)	Line Speed (fpm)	Air Gap (in)	"Green" Bonds (gpi)				Failure Mode
					Op	C	Dr	Mean	
S7	on	570	1200	10	0.585	0.554	0.388	0.509	peel
S6	off	570	1200	10	0.01	0.009	0.01	0.010	peel
S8	on	570	1200	7	0.586	0.528	0.499	0.538	peel
S9	off	570	1200	7	0.017	0.015	0.008	0.013	peel
S14	off	595	1200	10	0.663	0.517	0.386	0.522	poly stretch to break
S15	on	595	1200	10	0.536	0.537	0.562	0.545	poly break
S16	on	595	1200	7	0.591	0.603	0.526	0.573	DNR
S17	off	595	1200	7	0.363	0.31	0.012	0.228	peel
S19	off	595	600	10	0.569	0.529	0.509	0.536	poly break
S35	off	550	600	10	0.009	0.008	0.008	0.008	peel
S36	on	550	600	10	0.599	0.621	0.577	0.599	poly break
R1	on	540	600	10	0.632	0.6	0.612	0.615	poly stretch & breaking
R3	on	575	1200	10	0.482	0.653	0.595	0.577	poly break
S39	off	540	600	7	0.009	0.007	0.008	0.008	peel
S40	on	540	600	7	0.613	0.542	0.629	0.595	poly stretch & breaking
S41	on	540	600	10	0.520	0.593	0.591	0.568	poly stretch
S42	on	530	600	10	0.594	0.588	0.561	0.581	poly stretch
S43	on	530	600	7	0.602	0.598	0.537	0.579	DNR
S44	on	510	600	7	0.619	0.442	0.547	0.536	Poly & break
S45	on	510	600	10	0.607	0.619	0.537	0.588	poly stretch

Melt Temperature: 313 C (595 F), Line Speed: 366 mpm (1200 fpm)  
**Compare Air Gaps**

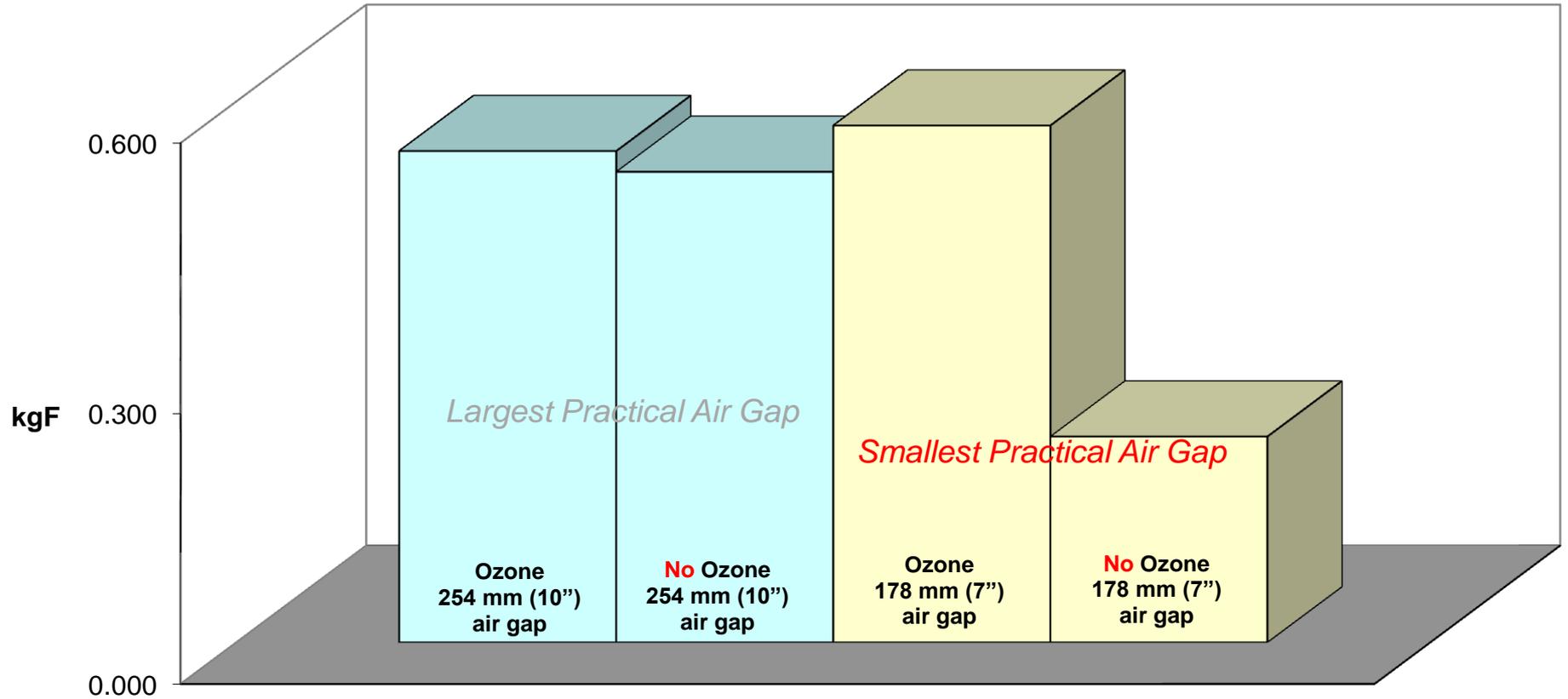


Melt Temperature: 313 C (595 F), Line Speed: 366 mpm (1200 fpm)



*Very Small Difference at 10 inch air gap because of adequate Air Gap*

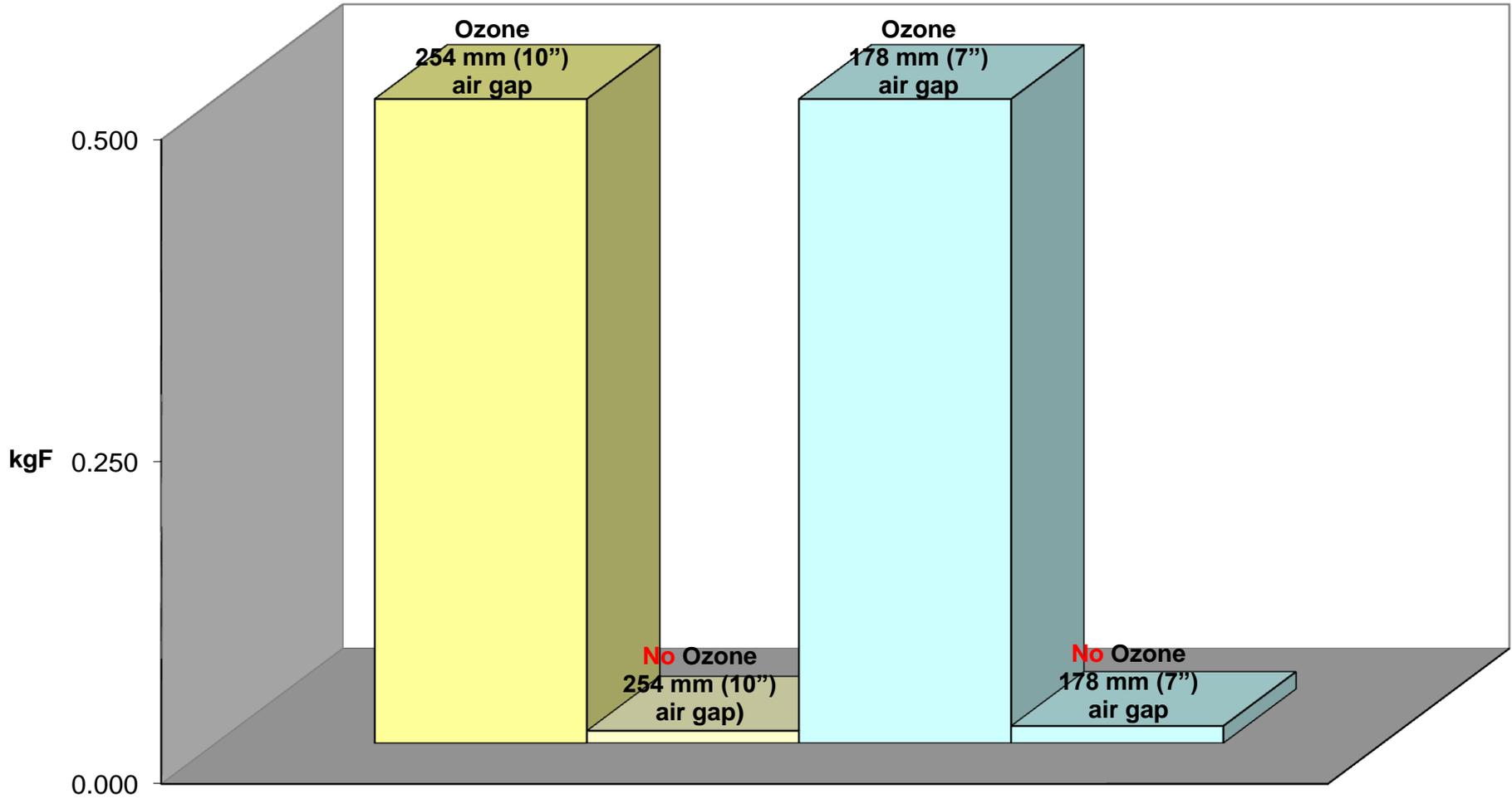
Melt Temperature: 313 C (595 F), Line Speed: 366 mpm (1200 fpm)



*Very Large Difference at 7 inch air gap because of inadequate Air Gap*

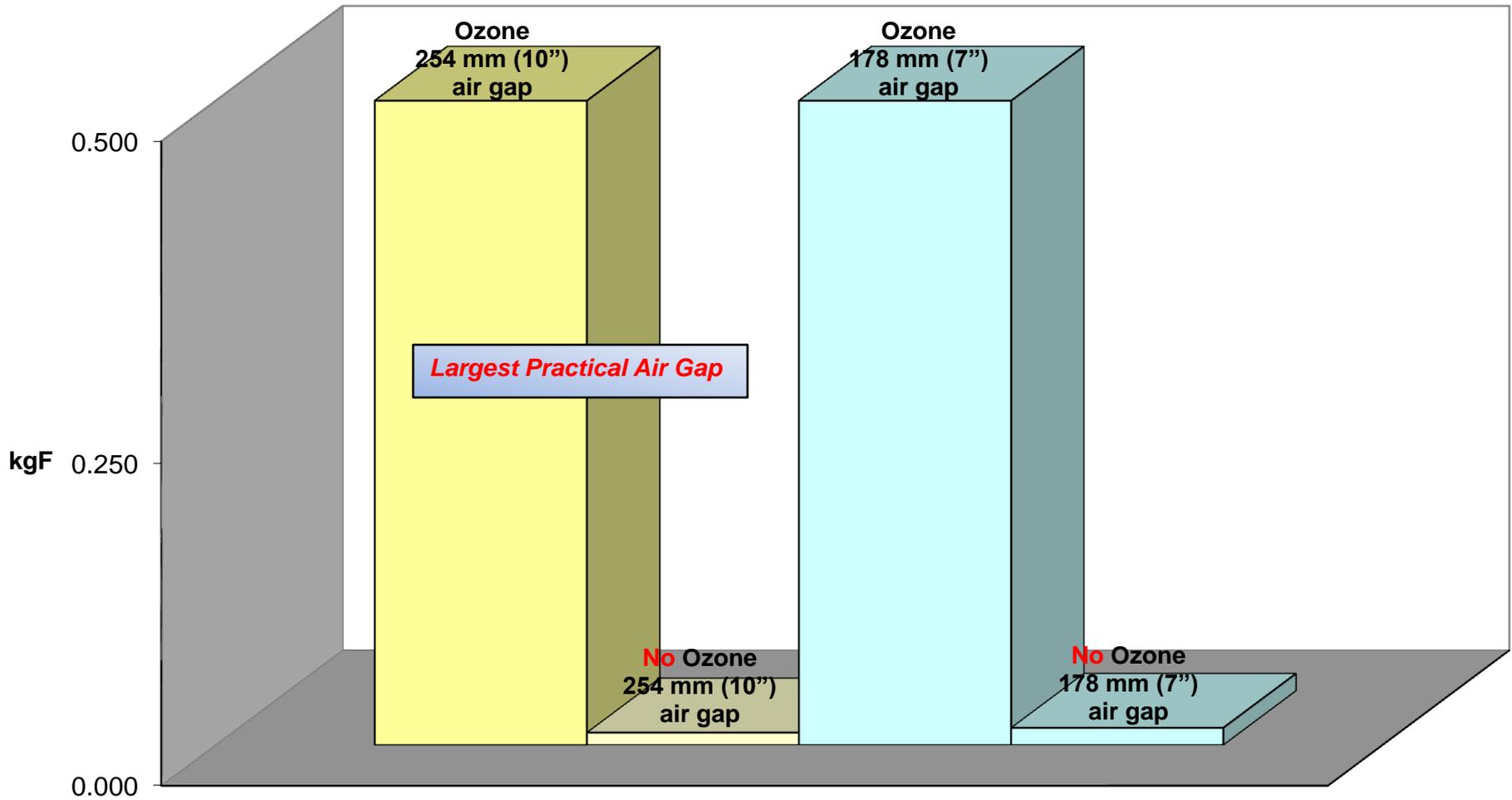
Melt Temperature: 299 C (570 F), Line speed: 366 mpm (1200 fpm)

**Compare Air Gaps with Ozone blanketing**



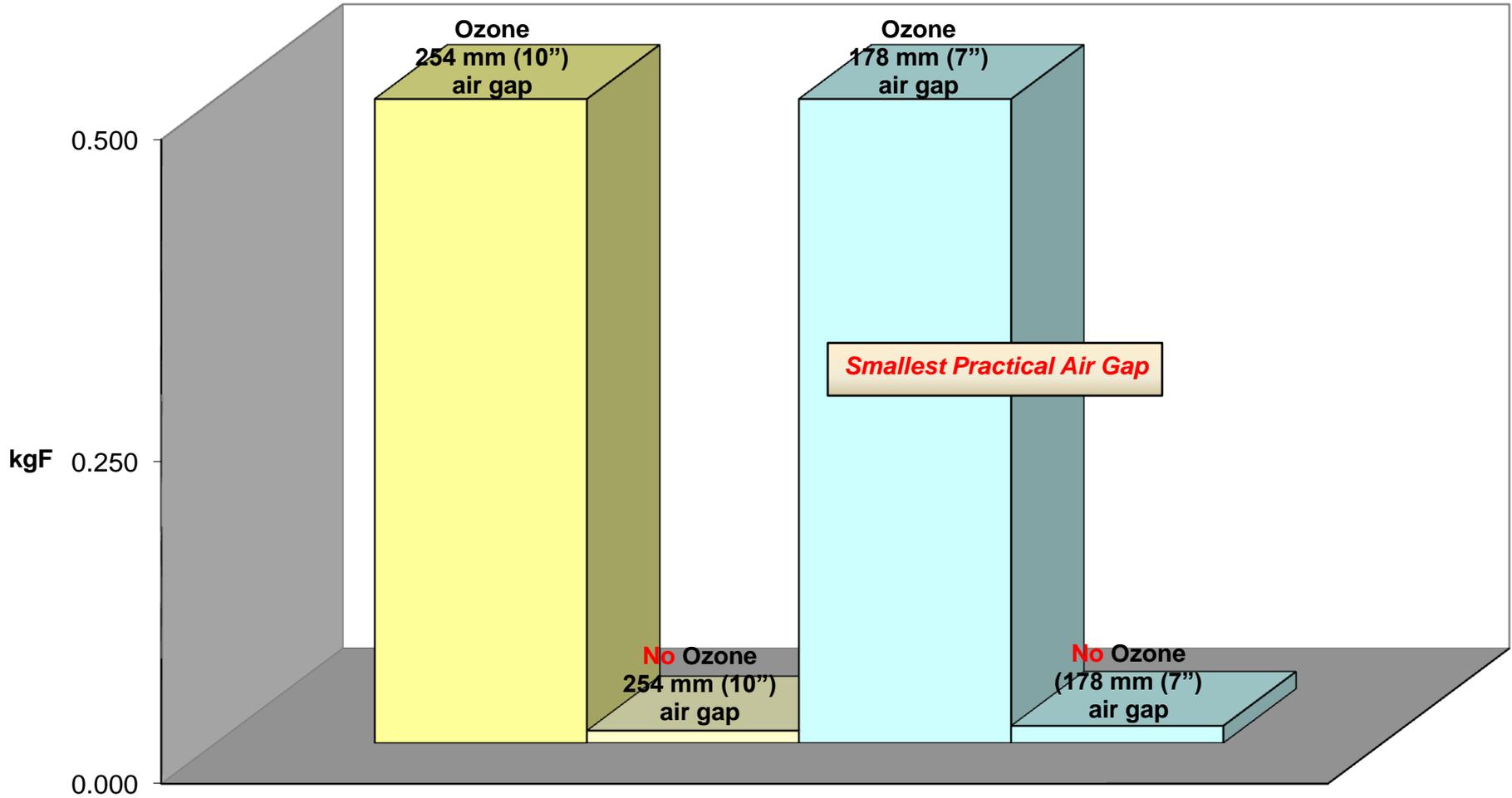
Melt Temperature: 299 C (570 F), Line speed: 366 mpm (1200 fpm)

**The Effect with Ozone Exposure**

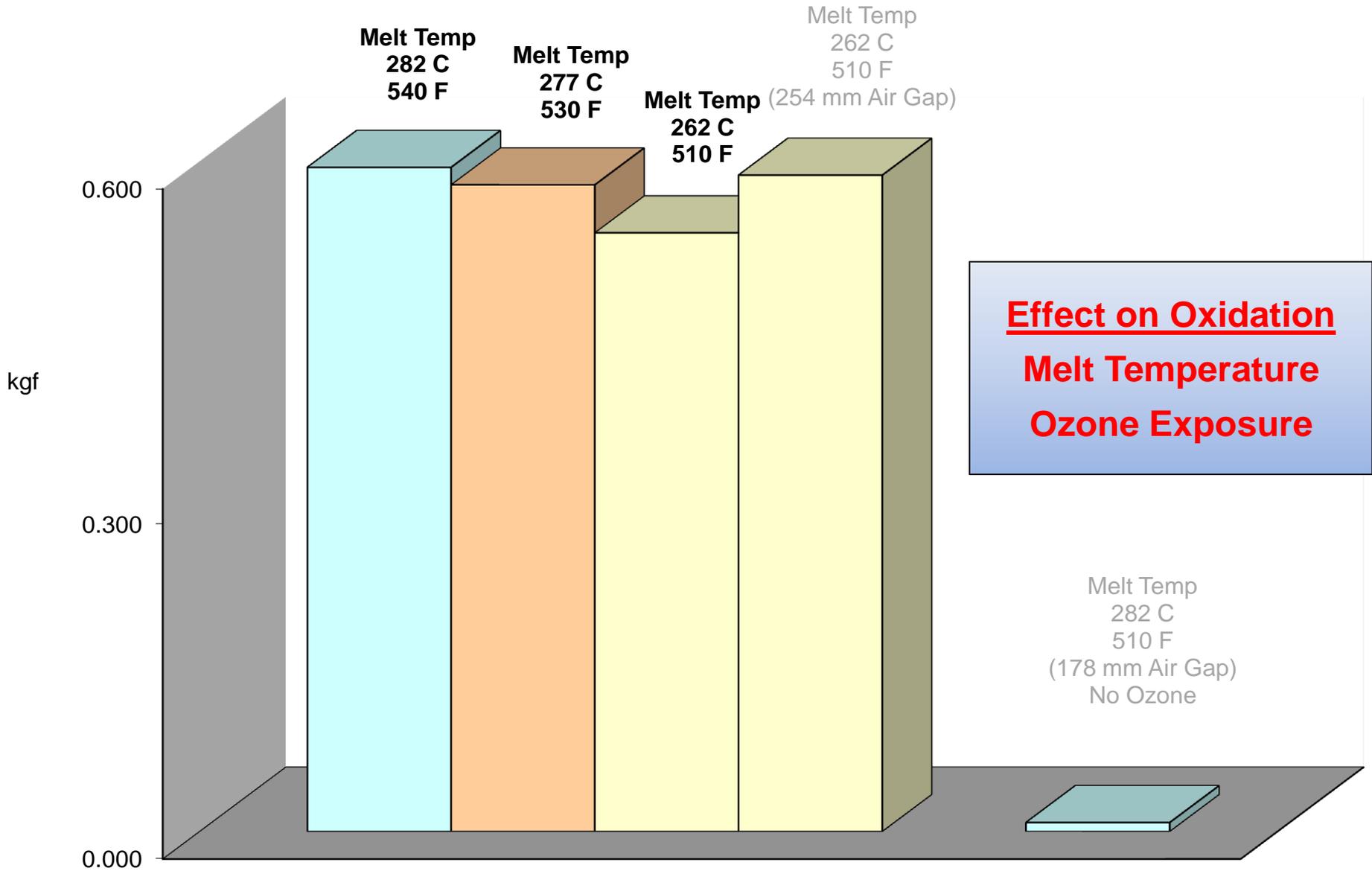


Melt Temperature: 299 C (570 F), Line speed: 366 mpm (1200 fpm)

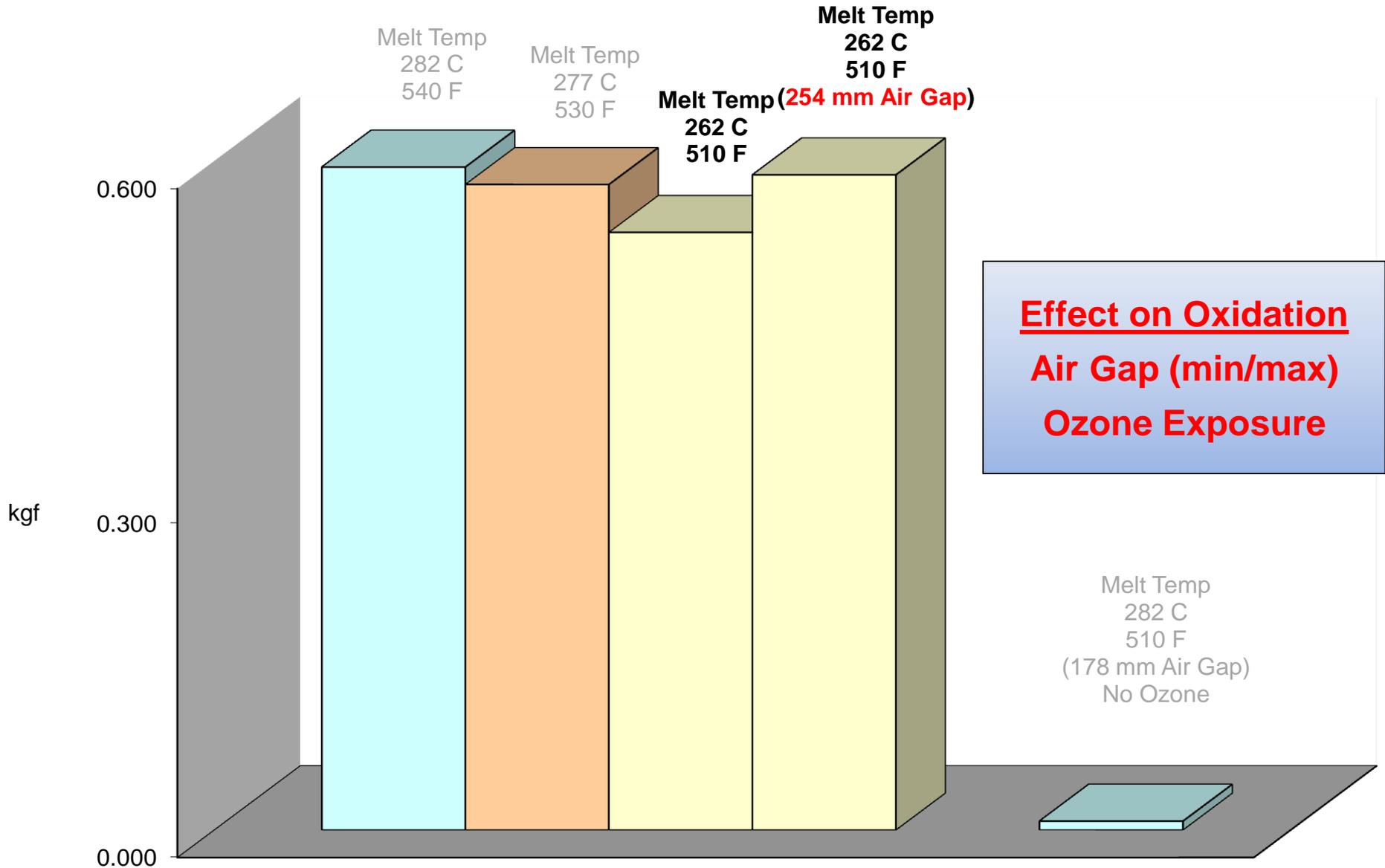
*Ozone Exposure has a tremendous effect on oxidation  
with a low temperature melt*



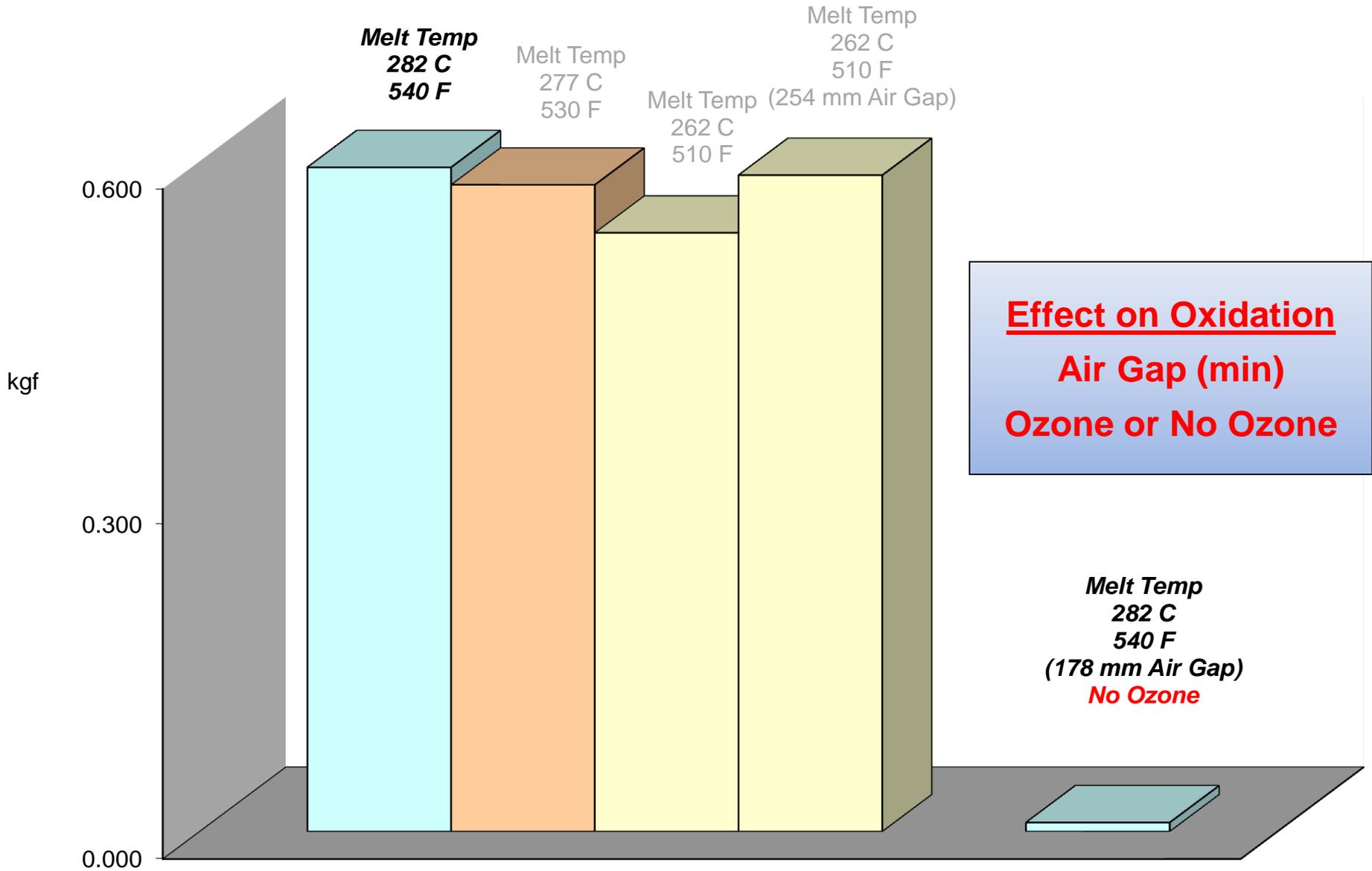
Air Gap: 178 mm (7") Line Speed: 183 mpm (600 fpm)

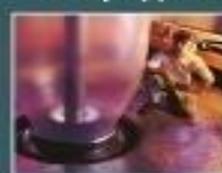


Air Gap: 178 mm (7") Line Speed: 183 mpm (600 fpm)



Air Gap: 178 mm (7") Line Speed: 183 mpm (600 fpm)





# Conclusions

In these experiments, with an extended run, it was demonstrated that with:

- ✓ Lowering the melt temperature by 14<sup>0</sup>C (57<sup>0</sup>F),
- ✓ Line speed @ 366 mpm (**1200** fpm),
- ✓ Air Gaps @ 178 mm (7") & 254 mm (10"),

An acceptable peel force can be achieved, with the use of ozone



# Conclusions

In these experiments, with an extended run, it was demonstrated that with:

- ✓ Lowering the melt temperature from 313<sup>0</sup>C (595<sup>0</sup>F) - 262<sup>0</sup>C (510<sup>0</sup>F),
- ✓ Line speed @ 183 mpm (**600** fpm),
- ✓ Air Gaps @ 178 mm (7") & 254 mm (10"),

Also has an acceptable peel force, with the use of ozone



# Conclusions

Samples gave the same peel force results with;

- reduction of melt temperature by 53<sup>0</sup>C (85<sup>0</sup>F)
- Air Gaps @ 178 mm (7”) and 254 mm (10”),
- ozone exposure

As the samples made with;

- proper TIAG,
- typical extrusion coating melt temperatures.

These parameter changes translate into cost savings on energy and the “wear” on the equipment.



# Conclusions

Also important is, *by reducing the temperature*, the polymer melt is not being degraded by excessive heat during the extrusion process .

The sealant layer is much more stable, less damaged.

This results in a vastly improved heat seal integrity.

Ozone blanketing when used in conjunction with Air Gap oxidation;

- can enhance oxidation at higher line speeds
- can enhance oxidation at lower melt temperatures

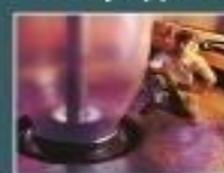


# Conclusions

The goal of these experiments was to use ozone as an aid to lower the extrusion melt temperature and help improve the seal integrity on heat seals.

This goal was accomplished while reducing the melt temperature by 53<sup>0</sup>C (85<sup>0</sup>F) and still maintaining the desired peel force.

Also demonstrated that a significant increase in line speed can be achieved without sacrificing bond performance or integrity.



# Future Experiments

Although these experiments have demonstrated the benefit of ozone exposure to the extrusion melt, there are more questions that need to be answered.

In future experiments, some of the variables could be;

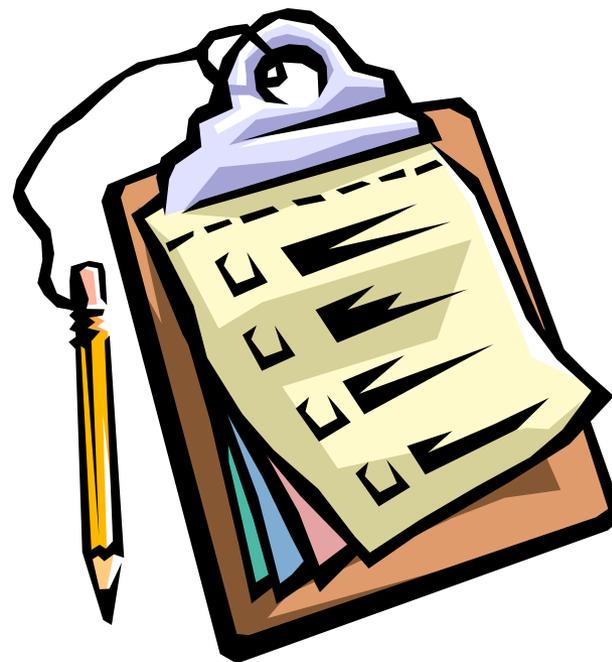
- Resins and resin blending,
- Ozone concentration, at different melt temperatures
- The level of oxidation measured directly on the sealant layer, such as FTIR analysis using the Carbonyl absorption band.
- Determine what is the dynamic relationship to varying the Line Speed, Melt Temperature and Air Gap (similar to a phase diagram) .



*Thank You for your  
kind attention.*

**PRESENTED BY**

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*Please remember to turn in  
your evaluation sheet...*